An Industrial Fluid Multi-Sensor

Nicholas O. Liotta

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An Industrial Fluid Multi-Sensor

A Thesis Submitted in Partial Fulfillment of the
Requirements for the Degree of
Master of Science in Electrical Engineering

By
Nicholas O. Liotta

Approved by:

__________________________________________ Date_________________
Dr. Lynn F. Fuller (Thesis Advisor)

__________________________________________ Date_________________
Dr. Sergey E. Lyshevski (Thesis Committee Member)

__________________________________________ Date_________________
Dr. Ivan Puchades (Thesis Committee Member)

__________________________________________ Date_________________
Dr. Sohail A. Dianat (Chair Electrical and Microelectronic Engineering)

DEPARTMENT OF ELECTRICAL AND MICROELECTRONIC ENGINEERING
KATE GLEASON COLLEGE OF ENGINEERING
ROCHESTER INSTITUTE OF TECHNOLOGY
ROCHESTER, NEW YORK
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Abstract

Determining oil quality is an important part of any industry that uses oil as lubrication. Over time oil quality degrades with use or from contaminants being introduced. Since the majority of systems that use oil are closed systems there is no way to remove contaminants or recycle the oil to its original state. The only option is to change the oil. Even with frequent oil changes there is still a chance that a contaminant can be introduced, causing a failure. Currently there are sensors that test oil quality, but they are bulky compared to a Microelectromechanical (MEMs) multi-sensor. The objective of this project is to make a low cost industrial fluid multi-sensor to replace larger sensors that exists today. The industrial fluids multi-sensor will measure; oil quality, water in oil and temperature of the oil.

The quality of the oil will be determined by using Electrochemical Impedance Spectroscopy (EIS). This will be accomplished by using an interdigitated finger (IDF) electrodes to measure the impedance of the oil at different frequencies. With regular use and the introduction of other contaminants the resulting impedance changes, ultimately telling when your oil is old or contaminated and needs to be changed.

Water in oil will also be tested. An IDF capacitor with a polyimide coating will be used to determine the water in oil. Water in oil is important to sense because the oil does not lubricate as well if water is present. The polyimide absorbs the water, which changes the dielectric constant of the polyimide and increases the capacitance as water is added. The detection of water can be used as an early indication that there is a leak or a
broken gasket. Not fixing this problem can lead to a seized engine or non-uniform wearing of gears.

The temperature of the oil will be measured with a thin film resistor. The thin film resistor changes resistance as the temperature changes. Depending on the thermal coefficient of resistance (TCR), a greater or less change in resistance will occur for different materials.

Two revisions of the multi-sensor were designed, manufactured and tested at RIT. The EIS sensor and Temperature sensor were also tested by an outside company. One temperature sensor and 18 IDF capacitors made of tantalum were designed in Design 1. The IDF capacitors were used as either an EIS or water in oil sensor by adding polyimide. Design 1 had three major shortcomings that lead to Design 2. The first improvement was that all three sensors needed to put on one chip. Secondly, the nominal capacitance of the water in oil sensor needed to be increased. Lastly a different metal was needed for the temperature sensor because tantalum has either a positive or negative temperature coefficient of resistance. A new fabrication process was used to fabricate Design 2 to be able to get smaller widths and spaces of the IDFs. Two EIS, water in oil and one temperature sensor were designed in Design 2. All of the sensors in Design 2 were nickel on top of titanium. Quality of oil, water in oil and temperature were all able to be successfully tested using Design 2.
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Chapter 1: Introduction and Background

During a lubricant’s lifetime, a lubricant undergoes substantial chemical changes due to a variety of degradation mechanisms: oxidative high temperature degradation; contaminants by water glycol, fuel, soot, and wear metals [1]. It is difficult to predict when degradation will occur or a contaminant will be introduced to the system. Oil quality sensors need to be placed in the oil to check and be able to detect contaminants as well as degradation in the oil. Current methods used to determine oil quality, routinely performed by major engine and lubricant manufacturers, are frequent, repetitive and time consuming [1]. In order to make a low cost on-line oil quality sensor electrochemical impedance spectroscopy (EIS), water in oil and temperature sensing techniques will be used.

1.1 EIS Background

EIS techniques can be used whenever a voltage controlled transfer of electrical charge occurs at the interface between an electrode and an electrolyte [2]. This can be modeled by the equivalent circuit of a resistor and capacitor in parallel, Figure 1.1. The resistor, Re, is the resistance of the electrolyte and the capacitor, C, is the capacitance between the electrodes and the oil. The field lines of the capacitor go through the bulk of the oil. This allows a baseline measurement to be made when the oil is new and then a new test later to see the degradation of oil.
When the electrode is first placed in oil a frequency sweep is done as a baseline test before any contaminants or degradation occurs. A frequency sweep is done from 1 mHz to 10 MHz in [1] to demonstrate the impedance spectra or Nyquist plot of fresh and oxidized oil. Figure 1.2 shows the difference between fresh and oxidized oil at 120°C with an electrode spacing of 0.1 mm and 0.35 mm. The spacing of the electrode plays a role in the Nyquist plot because the length of the field lines going through the electrolyte changes. As the width of the spacing increases the resistance of the oil increase while the capacitance decreases. The wider spaced electrode gives a larger response shown in [1]. It is also shown in Figure 1.2 that as the oil oxidizes the magnitude of the impedance decreases. The impedance also changed at different temperatures, different electrode surface areas, electrode separation distance and electrochemical potential and degree of lubricant oxidative degradation [1]. Three different frequency ranges were used; low frequency range of 1 mHz to 100 mHz, medium frequency range of 100 mHz to 10 Hz and a high frequency range of 10Hz to 10MHz.
The low frequency range is affected by the spacing between electrodes and is measures the surface protective additives in the oil[1]. The medium frequency range is affected by the surface of the electrodes and is driven by the charge accumulation due to the absorption of the surface active lubricant additives[1]. The medium frequency gives data about the additives used in the oil. The high frequency impedance is a factor identified in the bulk of the solution being tested[1]. The high frequency range tells how the dielectric of the oil changes due to contaminants.
Interdigitated sensor arrays and parallel plate electrodes were also tested in [1] to see their different responses. The parallel plate electrodes were used to set a baseline for the other sensors tested. It was concluded that both interdigitated sensor arrays and parallel plate electrodes work to test oil quality.

Similar techniques used in [1] where used in [2] to determine the impedance of a system and some conclusions were made. First, a large range of frequencies need to be tested to determine the impedance of the oil. The high frequency limit gives the electrolyte’s resistance [2]. The impedance can be plotted to see the degradation of oil in a Nyquist plot in the complex plane. Lastly that the distance of the electrodes and temperature play a role in the impedance plot.

1.2 Water in Oil Background

Water in oil or moisture in oil sensing has been used in transformers to make sure that the electrical and mechanical properties do not deteriorate. It is well recognized that water in oil has detrimental effects on transformer performance as well as other applications where oil is used for lubrication. When the water in the oil exceeds the solubility for that temperature, free water will form [3]. When free water forms in oil, the oil does not act as a good lubrication any more. Water in oil testing is also good to see if there are any leaks or gasket failures because in a closed system water should not be introduced. Water in oil sensors have been made with a tube in tube design in [4]. The tube in tube water in oil sensor is shown in Figure 1.3. A tube in tube sensor’s
dielectric is the oil between the inner and outer electrode, as water is added to the oil its dielectric changes.

Water in oil can be tested both off- and on-line. Offline water in oil testing is done by analyzing a sample in a lab. On-line solution consist of either putting a water in oil sensor directly into the oil or making a separate path for the oil to travel through. Figure 1.4 shows an on-line water in oil testing setup where the oil has a direct path to go through a tube in tube sensor [5]. By having a specific track for the oil to go through samples of the oil can be taken to see if there is any water in oil. Tube in tube solutions are big and can be expensive to make and test the oil.

Parallel plate and IDF capacitor solutions have also been made for testing water in oil. A parallel plate capacitor with a dielectric of polyimide was tested in [5] as a water in oil sensor. When polyimide film capacitive humidity sensor is dropped into oil, there
is a moisture dynamic equilibrium between the polyimide film and transformer oil [5]. The relative dielectric of the polyimide changes as the water content in the oil changes because the amount of water absorbed by the polyimide increases. The change in capacitance was then translated through secondary circuitry to monitor the on-line water in oil content in [5]. Another structure that measures relative humidity and uses polyimide is an IDF capacitor.

In order to make a smaller, cost efficient humidity sensor an IDF relative humidity sensor was made in [6]. The sensor made in [6] can be seen in Figure 1.5. The IDF is covered with a polyimide film to increase sensitivity. Polyimide is a polymer particularly suitable as humidity sensitive layer thanks to its full compatibility with integrated circuit process, its great chemical stability, and its high permeability to water [6]. After the fabrication of an IDF capacitor the polyimide can be added at the end of the process. The polyimide layer absorbs the water which changes the dielectric of the polyimide.

![Figure 1.5: IDF Sensor Used in [6]](image)

One disadvantage to the sensor designed in [6] is the parasitic capacitances which appear between the interdigitated electrodes outside the polyimide film and affects the output signal but are independent of the humidity [6]. Figure 1.6 shows the parasitic capacitance of the sensor designed in [6]. In order to have a good humidity sensor the
parasitic capacitance needs to be reduced. Since the dielectric of silicon is roughly three times larger than the dielectric of polyimide, the main capacitance is coming from the substrate rather than the polyimide. This makes the substrate capacitance dominate the capacitance of the sensor. Similar to the test done in [5] the sensor built in [6] could be used as a water in oil sensor. The only difference would be how the capacitor is manufactured.

![Diagram of parasitic capacitance in an IDF sensor](image)

**Figure 1.6: Parasitic Capacitance Shown in IDF Sensor [6]**

### 1.3 Temperature Sensor Background

Temperature is an important parameter to monitor in oil. By knowing the temperature of oil a problem can be detected if the temperature is too high or low during operation. Temperature also needs to be monitored for other sensors to maintain accurate repeatable measurements.
Chapter 2: Theory

2.1 Electrochemical Impedance Spectroscopy (EIS)

An IDF capacitor sensor was designed to test oil quality using the EIS techniques. When the IDF is placed in the oil the system can be modeled as a capacitor and resistor in parallel shown in Figure 1.1. The real and imaginary parts of the impedance then are measured to see how the oil changes as contaminants are introduced. Figure 2.1 shows the geometry of the IDF where \( s \) is the size of the spaces, \( w \) is the width of the fingers and \( L \) is the length of the fingers.

![Figure 2.1: Geometry of IDF](image)

The capacitance can be found by using Equation 2.1 [7]. Equation 2.1 takes into consideration the number of fingers, \( N \), \( s \), \( w \), and \( L \). The dielectric of the oil is \( \varepsilon_r \) and the field lines, shown in Figure 2.2, are taken into consideration by the Bessel Thompson function. The capacitance from the glass and oil are added together in parallel. The
capacitance when glass is the dielectric accounts for 72% of the total capacitance and the oil accounts for 28%.

\[
C = LN4\varepsilon_0\varepsilon_r \pi n = 1 \times 12n - 1 Jo22n - 1\pi s2s + w
\] (2.1)

![Electric Field Lines](image)

**Figure 2.2: Electric Field Lines between IDF**

To better understand the relationship of capacitance and resistance in oil, a parallel plate capacitor was tested in oil. A parallel plate capacitor varies both capacitance and resistance, when in oil, by changing the area and spacing. Equation 2.2 shows the capacitance of a parallel plate capacitor.

\[
C = \rho \varepsilon_0 \varepsilon_r R
\] (2.2)

Equation 2.3 shows the relationship between the resistivity and resistance of the oil.

\[
R = \rho_{oil} A
\] (2.3)

By rearranging Equations 2.2 and 2.3 to equal A, the resistivity of oil, \( \rho_{oil} \), can be solved for.

\[
A = \rho_{oil} R = C s \varepsilon_0 \varepsilon_r
\] (2.4)
\[ \rho_{oil} = \frac{RC}{\varepsilon_0 \varepsilon r} = \frac{R}{\varepsilon_0 \varepsilon_r} \]

(2.5)

From Equations 2.4 and 2.5 the resistance of the oil can be rearranged to solve the resistance of the oil in terms of capacitance.

\[ R = \frac{\rho \varepsilon_0 \varepsilon_r C}{ \varepsilon_0 \varepsilon_r} \]

(2.6)

This relationship between capacitance and resistance of a parallel plate capacitor in oil holds true for IDF. By substituting the capacitance in Equation 2.1 into Equation 2.6 [1] the resistance of the oil can be rewritten as

\[ R = \frac{\rho \varepsilon_0 \varepsilon_r L N 4 \varepsilon_0 \varepsilon_r \pi n = 1 \infty 12 n - 1 J o 22 n - 1 \pi s 2 s + \omega}{1} \]

(2.7)

2.2 Water in Oil

The water in oil IDF use the same theory as the EIS, but a layer of polyimide is added on top of the IDF. The polyimide is added to increase the sensitivity to water in the oil. Instead of only using the dielectric constant of the oil and glass the dielectric constant of the polyimide and the dielectric constant of the water needs to be taken into consideration. The dielectric constant of the polyimide changes as water is added to the oil. The capacitance ratio of the capacitance, only due to the polyimide, with and without water can be seen in Equation 2.8 [6]. For quick approximations to see how the capacitance will change as water is absorbed Equation 2.8 can be used with the dielectric constant of polyimide ranging from 3.2 with no water, \( \varepsilon_{pwet} \), and 4 when no more water can be
absorbed by the polyimide, $\varepsilon_{\text{pdry}}$. $C_{\text{wet}}$ is the capacitance of the IDF when the polyimide has absorbed water.

$$C_{\text{wet}} = C_{\text{dry}} \varepsilon_{\text{wet}} \varepsilon_{\text{dry}}$$

(2.8)

To calculate the dielectric of the polyimide as water is absorbed the Empirical Looyenga formula [6], Equation 2.9, can be used to calculate $\varepsilon_{\text{pwet}}$. Where the $\gamma$ factor is the fractional volume of the water absorbed in the polyimide.

$$\varepsilon_{\text{pwet}} = \gamma \varepsilon_{\text{water}} 13 - \varepsilon_{\text{dry}} 13 + \varepsilon_{\text{dry}} 133$$

(2.9)

The water solubility of the oil also needs to be taken into consideration. Equation 2.10 gives an expression for the saturation solubility of water in oil, $r_s$, in ppm and temperature, $T$, in $^\circ\text{C}$ [3]. There is some discrepancy for the coefficients A and B. Table 2.1 shows the different coefficient values from different authors. Table 2.2 shows the data gathered by the different authors to describe the solubility of water in oil at different temperatures.

$$r_s = eA - BT$$

(2.10)

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Oommen</th>
<th>Griffin</th>
<th>Shell</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>7.42</td>
<td>7.09</td>
<td>7.3</td>
</tr>
<tr>
<td>B</td>
<td>1670</td>
<td>1567</td>
<td>1630</td>
</tr>
</tbody>
</table>

Table 2.1: Different Values for Coefficients in Equation 2.10 [3]

<table>
<thead>
<tr>
<th>$T$ ($^\circ\text{C}$)</th>
<th>Oommen</th>
<th>Griffin</th>
<th>Shell</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>20</td>
<td>23</td>
<td>22</td>
</tr>
<tr>
<td>10</td>
<td>33</td>
<td>36</td>
<td>35</td>
</tr>
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<td>30</td>
<td>82</td>
<td>83</td>
<td>84</td>
</tr>
<tr>
<td>40</td>
<td>122</td>
<td>122</td>
<td>124</td>
</tr>
<tr>
<td>50</td>
<td>179</td>
<td>174</td>
<td>180</td>
</tr>
</tbody>
</table>
The data in Table 2.2 was plotted in Figure 2.3. The average was taken of all of the data and an average expression for $r_s$ was found. A new expression can now be used to calculate $r_s$, shown in Equation 2.11.

$$rs_{avg}=25.834e^{0.0363T}$$ (2.11)
There is a linear relationship between the relative humidity of the oil and the moisture content of the oil, $r$, shown in Equation 2.12 [3], where $R_H$ is the percent relative humidity (%).

$$r = rsavgRH100$$

(2.12)

### 2.3 Temperature Sensor Theory

The resistance of a thin film resistor can be found by knowing the resistivity, $\rho$, length, $L$, width, $w$, and thickness, $t$, of the metal. Figure 2.4 shows the geometry of the metal resistor. The resistance, $R$, can then be found by Equation 2.13.

![Figure 2.4: Geometry of a Metal Resistor](image)

$$R = \rho L wt$$

(2.13)

As temperature changes, the resistance of a metal resistor also changes. The change in resistance is due to the thermal coefficient of resistance, $\alpha$, is usually positive for metals. Tantalum thought can have wither a positive or negative temperature coefficient of resistance. Figure 2.5 shows the change in resistance of a nickel resistor.
with temperature. The change in resistance due to $\alpha$ and the change in temperature, $T$, is shown in Equation 2.14 [8]. $R_T$ is the new resistance at temperature, $T_2$. The original temperature, $T_1$ is also needed to calculate the change in temperature. Although $\alpha$ has units of ppm/$^\circ$C, a change in temperature can result in a voltage change in the mV or higher range, depending on the current.

$$R_T = R + \alpha R(T_2 - T_1)$$  \hspace{1cm} (2.14)
Chapter 3: Design

Two revisions of a MEMS multi sensor were designed to include EIS, water-in-oil and temperature. A glass substrate was chosen for both revisions instead of a silicon substrate to reduce parasitic capacitance, as seen in [6]. The advantage to a glass substrate is the EIS and water in oil sensors are more sensitive to changes in the oil because glass has a lower dielectric constant than silicon. The dielectric constant of glass is 3.9 and the dielectric of oil is roughly 1.5, depending on the oil. This means that the oil contributes to 28% of the capacitance. If silicon was used as the substrate the oil would only contributes to 11% of the capacitance since silicon’s dielectric constant is 11.7 and the oil would only account for 11% of the change in capacitance. A glass substrate will allow the EIS and water-in-oil to be more sensitive to contaminants, degradation of the oil and water in oil.

3.1 Design 1

The die size for Design 1 was 5mm x 5mm, which allowed eighteen different designs to fit on one mask. There were eighteen different IDF designs and one temperature sensor designed. A full factorial design of experiments was done by changing the $w$ and $s$ of the IDF and keeping $L$ constant, Table 3.1. $N$ was designed to be the maximum number of $w$ and $s$ that fit with in the area given and was the overall length $l$ divided by $L$. The size of the $w$ and $s$ were designed to be 5µm, 12.5µm or 20µm, which allowed for a range of capacitances from 15pF to 68.5pF to be manufactured. The
capacitance was constrained by the dimensions of the chip, a larger area would allow for higher capacitances. The IDF were designed to be used for either EIS or water in oil sensors depending on the size of $w$ and $s$. The smaller $w$ and $s$ were designed to be the water in oil sensors because they would have a larger change in capacitance resulting from water added to the oil. The larger $w$ and $s$ were designed to be EIS sensors. But all of the IDF designs can be used for either EIS or water in oil sensors. The only difference is that to detect water in oil, the IDF are coated with a thin layer of polyimide. The thin film resistor was designed to be $6k\Omega$ on all chips. Table 3.2 shows the design of the temperature sensor. The layout for Design 1 sensors is shown in Figure 3.1. Each chip has one temperature sensor and one set of IDF. All of the sensors were designed to be tantalum because tantalum does not corrode in oil and the same processing steps could be used for all sensors.

<table>
<thead>
<tr>
<th>Device</th>
<th>s (um)</th>
<th>w (um)</th>
<th>l (m)</th>
<th>L (um)</th>
<th>N</th>
<th>C_{glass} (pF)</th>
<th>Coil (pF)</th>
<th>C (pF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>5</td>
<td>0.75</td>
<td>4390</td>
<td>157</td>
<td>23.44</td>
<td>9.01</td>
<td>32.45</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>5</td>
<td>1</td>
<td>4390</td>
<td>209</td>
<td>31.25</td>
<td>12.02</td>
<td>43.27</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>12.5</td>
<td>0.5</td>
<td>4375</td>
<td>105</td>
<td>21.49</td>
<td>8.27</td>
<td>29.76</td>
</tr>
<tr>
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<td>5</td>
<td>12.5</td>
<td>1.27</td>
<td>4375</td>
<td>241</td>
<td>49.47</td>
<td>19.03</td>
<td>68.49</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
<td>20</td>
<td>0.5</td>
<td>4360</td>
<td>105</td>
<td>24.99</td>
<td>9.61</td>
<td>34.60</td>
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<tr>
<td>6</td>
<td>5</td>
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<td>37.48</td>
<td>14.41</td>
<td>51.89</td>
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<tr>
<td>7</td>
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<td>5</td>
<td>0.5</td>
<td>4390</td>
<td>104</td>
<td>11.37</td>
<td>4.37</td>
<td>15.74</td>
</tr>
<tr>
<td>8</td>
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<td>0.75</td>
<td>4390</td>
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<td>17.06</td>
<td>6.56</td>
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<tr>
<td>9</td>
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<td>0.3</td>
<td>4375</td>
<td>63</td>
<td>9.37</td>
<td>3.60</td>
<td>12.98</td>
</tr>
<tr>
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<td>12.5</td>
<td>0.6</td>
<td>4375</td>
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<td>18.74</td>
<td>7.21</td>
<td>25.95</td>
</tr>
<tr>
<td>11</td>
<td>12.5</td>
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<td>0.45</td>
<td>4360</td>
<td>95</td>
<td>16.60</td>
<td>6.38</td>
<td>22.98</td>
</tr>
<tr>
<td>12</td>
<td>12.5</td>
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<td>0.65</td>
<td>4360</td>
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<td>22.65</td>
<td>8.71</td>
<td>31.36</td>
</tr>
<tr>
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<td>5</td>
<td>0.35</td>
<td>4390</td>
<td>73</td>
<td>6.85</td>
<td>2.64</td>
<td>9.49</td>
</tr>
<tr>
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<td>0.5</td>
<td>4390</td>
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<td>9.79</td>
<td>3.77</td>
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<td>4375</td>
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<td>9.27</td>
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<td>12.83</td>
</tr>
<tr>
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<td>0.6</td>
<td>4375</td>
<td>122</td>
<td>15.42</td>
<td>5.93</td>
<td>21.35</td>
</tr>
<tr>
<td>17</td>
<td>20</td>
<td>20</td>
<td>0.3</td>
<td>4360</td>
<td>63</td>
<td>9.37</td>
<td>3.60</td>
<td>12.97</td>
</tr>
</tbody>
</table>
Table 3.1: Design 1 of EIS or Water in Oil Sensor

<table>
<thead>
<tr>
<th></th>
<th>s (um)</th>
<th>w (um)</th>
<th>t (um)</th>
<th>N</th>
<th>rho (uΩ/cm)</th>
<th>l (um)</th>
<th>L (um)</th>
<th>R (Ω)</th>
</tr>
</thead>
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<td>20</td>
<td>0.4</td>
<td>10</td>
<td>120</td>
<td>3800</td>
<td>38100</td>
<td>5720</td>
</tr>
</tbody>
</table>

Table 3.2: Design 1 of Temperature Sensor

Table 3.1: Design 1 of EIS or Water in Oil Sensor

3.2 Fabrication of Design 1 Multi Sensor

Design 1 of the multi sensor was fabricated at R.I.T. during the summer of 2011. Fabrication began with a 6 inch glass wafer. Figure 3.2 shows the cross sectional view of the glass wafer before fabrication.

The first step was to deposit tantalum (Ta) onto the wafer using the CVC 601. The wafers are first loaded into the CVC 601 and the chamber is pumped down until the
base pressure is below $1 \times 10^{-5}$ Torr. A pre-sputter was done for 5 minutes with a radiant heat at 200°C. Then a 2 minute sputter of TaN (9% $N_2$) was done with power at 175 watts, 5 mTorr of Ar and .5 mTorr $N_2$. This was immediately followed by a Ta sputter with a power of 200 watts, 5.5 mTorr of Ar for 30 minutes. Figure 3.3 shows the cross sectional view after depositing Ta. The resulting Ta was 2500Å thick and was alpha-Ta. Beta-Ta was also fabricated using a higher power of 500 watts.

The problem with getting a different Ta is they have different temperature coefficients and the signal processing needs to account for whether or not the change in resistance will change the voltage either positively or negatively. Alpha-Ta has a positive temperature coefficient where beta-Ta has a negative temperature coefficient. It was believed that the variation in thickness would only have an effect on the difference in resistance and not have an affect on the temperature coefficient (see section Testing Temperature Sensor Design 1 for more details).

Following the Ta sputter, the wafer was then coated with photoresist on the SSI track one using coat.rcp. Coat.rcp is a three step process which starts which a dehydrate bake at 140°C with a HMDS vapor prime for 60 seconds. The wafer is then spin coated with OIR 620-10 resist at 3250 rpm for 30 seconds followed by a soft bake at 90°C for 60 seconds. Figure 3.4 shows the cross sectional view of the after coating with photoresist.
The photoresist was then exposed using the ASML Stepper with an exposure dose of 250mJ/cm²; see Figure 3.5 for the mask design. The photoresist was then developed using the SSI Track 2 using develop.rcp. Develop.rcp begins with a post exposure bake of 110°C for 60 seconds. The exposure bake is followed by developing the resist with CD-26 developer for 50 seconds, a rinse of DI water for 30 seconds and a spin dry for 30 seconds at 3750 rpm. The last step of developing the photoresist is a Hard Bake at 140°C for 60 seconds. Figure 3.6 shows the cross sectional view of the wafer after the photoresist is exposed and developed.
After the photoresist was developed the Ta was then plasma etched using the LAM 490. Recipe FNIT1500 was used to etch the Ta in three steps. The first step’s chamber pressure is 260mTorr, a gap of 1.65cm, no power and 200sccm of SF$_6$ for 1 minute. The second step has the same parameter used in step 1, but the power is turned onto 125watts and there is endpoint detection or a max time of 30s. The last step’s chamber pressure is 260mTorr, a gap of 1.65cm, 125 watts power and 200sccm of SF$_6$ for a 40% over etch. Figure 3.7 shows the cross sectional view after the plasma etch.

The last step of fabrication is to strip the photoresist in acetone, rinse in DI water and then spin rinse dry. The final cross sectional view is shown in Figure 3.8. Upon completion of fabrication the wafer was inspected under a microscope to make sure there are not unwanted opens or shorts on the devices. Figure 3.9 shows the wafer after fabrication before the individual chips are cut using the wafer saw.
After fabrication the devices were cut using the wafer saw and the capacitance and resistance were checked. Some of the chips were hand painted with polyimide to turn the IDF into water in oil sensors.

Device #5 and #10 had shorts from manufacturing defects from the mask, so none of those chips worked. The devices with \( s=5\mu m \) had shorts on all the devices. This may be because the fingers were too close together that a small particle can short the device from the wafer saw or from some unknown factor. Only a couple of devices with \( s=12.5\mu m \) worked, but the majority had similar problems as with \( s=5\mu m \) and would be hard to consistently manufacture following this process. The majority of the devices with \( s=20\mu m \) worked. Figure 3.10 shows a manufactured multi sensor with \( w=20\mu m \) and
$s=20\mu m$. In order to consistently get smaller $w$ and $s$ a new process would need to be made.

![Manufactured Multi Sensor](image)

### 3.3 Design 2

A redesign was needed after testing Design 1. Design 2 needed to address the following improvements; all sensors need to be on one chip, increase the nominal value of the capacitance of the water in oil sensor, be able to solder to the chip and fix the crystal structure problem of Ta. All three sensors where integrated onto one 6.5mm x 6.5mm chip, Figure 3.11 shows the chip layout. This was accomplished by designing two different IDF, one for EIS and the other for water in oil.
The EIS sensor has similar \( w \) and \( s \) as Design 1. Two EIS sensor combinations were designed with different \( w \) and \( s \) because 20\( \mu \)m worked well in Design 1. An IDF with 15\( \mu \)m \( w \) and \( s \) was designed to give a larger capacitance. The EIS sensor was made with two different \( L \). The first \( L \) was made to fit between the EIS pads, the same as in Design 1, but the second \( L \) was designed to fit inside the pads of the temperature sensor. The second \( L \) is smaller than the first because it had to spaced away from the temperature sensor’s pads to avoid making electrical shorts. The total capacitance is the capacitance from the two different \( L \)s added together in parallel. This was done to optimize the length of the chip. Table 3.3 shows the EIS designs.

<table>
<thead>
<tr>
<th>EIS</th>
<th>( s ) (um)</th>
<th>( w ) (um)</th>
<th>( l1 ) (um)</th>
<th>( l2 ) (um)</th>
<th>( N1 )</th>
<th>( N2 )</th>
<th>( C_{\text{glass}1} ) (pF)</th>
<th>( C_{\text{glass}2} ) (pF)</th>
<th>( C_{\text{glass total}} ) (pF)</th>
<th>( \text{Coil} ) (pF)</th>
<th>( C_{\text{total}} ) (pF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>15</td>
<td>15</td>
<td>3970</td>
<td>3970</td>
<td>67</td>
<td>57</td>
<td>9.02</td>
<td>7.67</td>
<td>16.70</td>
<td>4.64</td>
<td>21.33</td>
</tr>
<tr>
<td>B</td>
<td>20</td>
<td>20</td>
<td>3960</td>
<td>3960</td>
<td>50</td>
<td>43</td>
<td>6.75</td>
<td>5.74</td>
<td>12.49</td>
<td>3.47</td>
<td>15.96</td>
</tr>
</tbody>
</table>

Table 3.3: Design 2 EIS Sensor
The water in oil sensor needed a larger nominal capacitance. This was achieved by decreasing the size of the \( w \) and \( s \) to either 2 or 3\( \mu \)m and increasing \( N \). Table 3.4 shows the two different water in oil designs. By increasing the nominal capacitance the change in capacitance will increase and will be easier to measure.

<table>
<thead>
<tr>
<th>Water in Oil</th>
<th>( s ) (um)</th>
<th>( w ) (um)</th>
<th>( l1 ) (um)</th>
<th>( l2 ) (um)</th>
<th>( N )</th>
<th>( C_{\text{glass}} ) (pF)</th>
<th>( C_{\text{polyimid}} ) (pF)</th>
<th>( C_{\text{total}} ) (pF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2</td>
<td>2</td>
<td>3996</td>
<td>3996</td>
<td>500</td>
<td>68.13</td>
<td>26.20</td>
<td>94.33</td>
</tr>
<tr>
<td>B</td>
<td>3</td>
<td>3</td>
<td>3994</td>
<td>3994</td>
<td>333</td>
<td>45.40</td>
<td>17.46</td>
<td>62.86</td>
</tr>
</tbody>
</table>

Table 3.4: Design 2 Water in Oil Sensor

Soldering to the pads instead of wiring bonding became a priority for packaging, so the metal was changed from Ta to a Titanium/Nickel (Ti/Ni). Originally a layer of Ni was going to be sputtered ontop of the Ta, but Ni can not be plasma etched. The Ta crystal problem would also still not be resolved. Another problem with plasma etching is it does not consistently etch the small \( w \) and \( s \). So by decreasing the size of \( w \) and \( s \) for the water in oil sensor a new manufacturing process is needed. Switching the metal to Ti/Ni also solved the alpha/beta crystal structure problem that gives two different thermal coefficients of resist, one positive and the other negative. By changing the metal, the temperature sensor became easier to manufacture reliably. The overall resistance of the temperature sensor was also increased to achieve a bigger change in voltage with temperature change. Table 3.5 shows the design of the temperature sensor. There were four different combinations designed in Design 2. All combinations for the EIS sensor and water in oil sensor were manufactured with the temperature sensor.

<table>
<thead>
<tr>
<th>Temp</th>
<th>( s ) (um)</th>
<th>( w ) (um)</th>
<th>( t ) (um)</th>
<th>( N )</th>
<th>rho (( \mu )( \Omega )/cm)</th>
<th>( l ) (um)</th>
<th>( L ) (um)</th>
<th>( R ) (( \Omega ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>5</td>
<td>0.15</td>
<td>20</td>
<td>9.5</td>
<td>4190</td>
<td>83900</td>
<td>10627</td>
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</table>

Table 3.5: Design 2 Temperature Sensor
3.4 Fabrication of Design 2 Multi Sensor

The process to manufacture Design 2 had to be changed because of the smaller \( w \) and \( s \) of the water in oil sensor and using a different metal, Ti/Ni. Both the smaller \( w \) and \( s \) was too difficult to with RIT’s processing capabilities. By changing the feature size and the metal a new process was used to manufacture Design 2 that utilizes a negative photoresist, evaporation and lift-off.

Design 2 of the multi sensor was fabricated at R.I.T. during the winter of 2011-2012. Fabrication began with a 6 inch glass wafer, see Figure 3.3. The first step of processing was a piranha clean. The piranha clean is used to remove organic materials that could have formed on the glass substrate. A piranha clean is a 1:1 solution of Sulfuric Acid (\( \text{H}_2\text{SO}_4 \)) and Hydrogen Peroxide (\( \text{H}_2\text{O}_2 \)) that is heated to 130\(^\circ\)C. The wafer is then placed in the solution for 15 minutes. The piranha clean is followed a DI rinse for 5 minutes and a spin, rinse, dry. Once the wafer is dry the wafer is ashed using the GaSonic Asher. An oxygen plasma etch was done for 198 seconds using Recipe FF on the GaSonic Asher. After being ashed the wafer is ready to be coated with photoresist.

The wafer was coated using SSI Track 1 recipe NLOFCOTG. First the wafer underwent a dehydration bake with HMDS Vapor prime at 140\(^\circ\)C for 3 minutes and was followed by a 30 second cool. AZ nLOF 2020 negative photoresist was then hand coated on the wafer at 2500 rpm for 60 seconds. A soft bake at 110\(^\circ\)C for 3 minutes followed. Figure 3.12 shows the wafer coated with photoresist after going through SSI Track 1.
The photoresist was then exposed using the ASML Stepper with an exposure dose of 66mJ/cm²; see Figure 3.13 for the mask used. The mask had two defects on it; one was a short one of the EIS sensors and water in oil sensors. The photoresist was then developed using the SSI Track 2 using DEVNLOFG.rcp. DEVNLOFG.rcp begins with an Image Reversal Bake of 110°C for 3 minutes followed by a 30 second cool. Then the photoresist is developed with CD-26 developer for 5 seconds while spinning at 50rpm. Then more CD-26 is dispensed for 5 seconds and the photoresist is developed for 70 seconds. After the developer a rinse of DI water is done for 30 seconds at 1000rpm. This is followed by a spin dry for 30 seconds at 3750 rpm. Figure 3.14 shows the cross sectional view of the wafer after the AZ nLOF 2020 is exposed and developed.
Once the photoresist is developed the wafer is ready for evaporation. Evaporation of the Ti/Ni was chosen over sputtering because the lift-off was difficult to do and the small features had shorts or were gone altogether. One advantage of sputtering is it can hide changes in height. This means that when the Ti/Ni was sputtered on to photoresist there was not a gap between the metal on top of the photoresist and the metal on the substrate, making it difficult to lift-off. Figure 3.15 shows IDF after an unsuccessful lift-off with sputtered Ti/Ni. Notice that not all the spaces are cleared thus shorting the two electrodes.

The CVC evaporator was used to evaporate the Ti and Ni onto the wafer. The Ti acts as an adhesion layer between the glass and the Ni. Before evaporation, one pellet of Ti, .110 grams, was placed in a woven tungsten (W) basket and placed into the CVC Evaporator. The wafer is then placed 25cm directly above the W basket, this gives a
thickness of the deposited Ti to be \(\sim 300\text{Å}\). The CVC evaporator will allow up to four different metals to be evaporated during one run of the machine. Two pellets of Ni, .218 grams each, are placed into separate woven W baskets and are put in the next available position in the CVC evaporator. One pellet of Ni gives a thickness of \(\sim 300\text{Å}\) at 25cm away from the W basket. After the Ti is deposited the operator must move the first Ni pellet into position to be evaporated and move the second Ni pellet into position after the first Ni pellet is evaporated.

Once the W baskets are loaded with the correct metal, the bell jar of CVC evaporator needs to be closed and the chamber will begin to pump down. Before evaporating the Ti, the chamber needs to be pumped down to \(2 \times 10^{-6}\text{Torr}\). The Ti can then be evaporated by turning on the filament and slowly increasing the power to 220-240V. The Ti will begin to evaporate around 220V. The power was kept between 220V and 240V until the Ti was completely evaporated. Figure 3.16 shows the cross sectional view of after the Ti was evaporated.

![Figure 3.16: Cross Section of Design 2 after Ti Evaporation](image)

After the Ti was fully evaporated the first Ni pellet is moved into position to be evaporated. After the chamber has pumped back down to \(2 \times 10^{-6}\text{Torr}\) the Ni can be evaporated. The filament was ignited and the power was slowly increased to 200V. Around 200V the Ni begins to evaporate. The power was kept between 200V and 220V.
until the Ni was completely evaporated. The second pellet of Ni is evaporated the same way as the first after the chamber has pumped down again. Figure 3.17 shows the cross sectional view wafer after both Ni pellets are evaporated. The two pellets are evaporated separately because when evaporated at the same time, not all of the Ni is evaporated. The Ni does not fully evaporate because the W basket usually breaks before the evaporation is complete. The pressure in the chamber also increases because the evaporation takes a longer amount of time and more power than evaporating the pellets separately.

![Ni](image)

**Figure 3.17: Cross Section of Design 2 after Ni is Evaporated**

The last process step for fabricating Design 2 was to remove the photoresist and unwanted metal through lift-off. Lift-off was done using the Ultrasonic Wet Bench.

A square glass dish was placed with the bottom of the dish in the DI water of the Ultrasonic Wet Bench. Half an inch of acetone was poured into the glass dish. The ultrasonic was turned on and the wafer was placed in the acetone. While the Ti/Ni is being lifted off a magnet was used to pick up all loose metal. The lift-off takes a couple of minutes. After all the unwanted metal is lifted-off the wafer was rinsed in DI water. Figure 3.18 shows the cross sectional view wafer after lift-off.

![Figure 3.18: Cross Section of Design 2 after Lift-Off](image)
The wafer was inspected under a microscope to make sure that the lift-off was complete. Figure 3.19 shows a section of all three finished sensors water in oil sensor. Visually, most of the sensors looked good. The wafer was cut using the wafer saw before being tested. Polyimide was painted on to the water in oil sensor.

![Figure 3.19: Parts of All Sensors under 2.5X Microscope](image-url)
Chapter 4: Packaging and Test Setup

In order to test the sensors, packaging, testing procedures and signal conditioning was created to test each sensor. After the wafers were diced each chip needed to be packaged before being able to be tested. In order to test each sensor, three separate tests were created with their own circuitry. Labview™ was used to test the EIS and Temperature sensors, while an Arduino was used to test the Water in Oil sensor. Each test had its own signal conditioning and procedure for testing. Each circuit was then placed onto a single PCB, shown in Figure 4.1, for final testing.

Figure 4.1: Final PCB for Signal Processing

4.1 Packaging

Before packaging any chips, they were checked under the microscope and probed with fine tipped wafer probes. They were then connected LCR meter to check for opens and shorts. The LCR meter showed whether or not the EIS or water in oil sensor had shorts. It did this by checking the impedance, or capacitance and resistance, of the
sensor. For the capacitance sensors, the phase needs to close to -90 degrees. This ensures that the device is purely a capacitor. Some devices had both a capacitance and resistance. These devices were not packaged because the sensor will not work as designed. Occasionally a device will have a phase of 90 degrees and have a short circuit, but will show up on the LCR meter as an open. To double check that there is not any resistance, resistance is measured using a multi-meter. The multi-meter should read overload, which indicate that there is an open. When checking the capacitance sensors the resistance must be zero for them to function properly. If the resistance was not zero then that device was not packaged. The temperature sensor was also checked using the LCR meter, but since it is resistor the LCR meter measured the resistance and made sure there was not an open. The impedance was also measured and the phase was 90 degrees. After each sensor on a chip was checked using the LCR meter the water in oil sensor needed have polyimide painted onto the IDF.

Before coating the polyimide, the chip was cleaned with acetone and isopropyl alcohol. The polyimide used was HD Microsystems PI-2556 and is painted onto the water in oil sensors’ IDF. The polyimide is applied by taking a wire and dipping it into the polyimide so a drop of polyimide is on the tip of the wire. The polyimide is then applied on the IDF until all the fingers are covered. It is important not to cover the pads with polyimide because it is difficult to wire bond through polyimide. If one drop did not cover the entire fingers a second drop should be applied carefully not to cover the other sensors. Once the polyimide has been applied it needs to be cured in the oven for 30 minutes at 200°C. It is important to cure the polyimide before epoxying the chip onto the
PCB because the epoxy will melt at 200°C. Figure 4.2 show a chip from Design 1 and Design 2 after the polyimide has been applied. After the polyimide has been cured the chip or chips, Design 1, are ready to be packaged.

Figure 4.2: Polyimide applied to Design 1 (Left) and Design 2 (Right)

Copper PCBs were designed using expressPCB software and were manufactured at RIT. A different PCB was designed for each sensor because of the different sizes and pad placement. Design 1 needed two chips to be placed on one PCB to be able to measure EIS, water in oil, and temperature since each chip sensed EIS or water in oil. Since design 2 incorporates all sensors on one chip, only one chip is needed. The chips were epoxied to the PCB and wire bonds were used to connect the pads to the PCB. Epoxy was then used to cover the wire bonds, to prevent them from breaking during use and cleaning. Figure 4.3 shows the packaging for Design 1.
The pin out for both designs is exactly the same so the same cables can be used to test both devices. The outside pins are for water in oil, the second pins in from each side is the temperature sensor and the middle two pins are for EIS. As long as the correct pins are hooked up it does not matter which pin is in or out, they are interchangeable.

4.2 EIS Test Setup

In order to test oil quality using the EIS sensor, frequency analysis and the impedance of the EIS sensor in parallel with the resistance of the oil needs to be measured. A Labview™ routine was created to test the sensors. Before testing the devices the signal conditioning circuit for EIS Testing, Figure 4.4, was simulated in MATLAB. The transfer function of the signal conditioning circuit was found by first finding the impedance of the EIS sensor, which is shown in Equation 4.1.
This was then combined in parallel with the resistance of the oil, Equation 4.2, to find the impedance at the input of the op-amp, \(Z_1\).

\[
Z_1 = \frac{1}{R_\text{oil}} + sCEIS - 1 = \frac{1}{R_\text{oil}} + sCEIS R_{oil}
\]

(4.2)

The impedance of the output, \(Z_2\), is just the resistor \(R_2\) is shown in Equation 4.3. \(R_2\) was chose to be 1M\(\Omega\) to magnify voltage change at \(V_1\).

\[
Z_2 = R_2
\]

(4.3)

The transfer function is then found by dividing the impedance of the output by the impedance of the input times negative one to account for the inverting amplifier.
Equation 4.4 shows the transfer function of the signal conditioning circuit used for EIS Testing.

\[ H_s = -\frac{Z_2Z_1}{R_2R_{oil}^1 + sCEISR_{oil}} = -\frac{R_2R_{oil}^1 + sCEISR_{oil}}{R_2R_{oil}^1} \]

(4.4)

The transfer function in Equation 4.4 was simulated using MATLAB to better understand what the output of the EIS testing would be when a frequency sweep was done at the input, \( V \). Figure 4.5 shows the bode plot simulated in MATLAB. The bode plots of the EIS signal conditioning circuit has one pole when modeled by capacitor and resistor in series. The magnitude continues to increase after the pole. The simulated bode plot was done with the \( C_{EIS} = 90\text{pF} \) and \( R_{oil} = 20\text{M}\Omega \). The phase is dominated by the capacitance of the EIS sensor and there is a 90 degree change beginning a decade before the pole at 555 rad/s and continues until a decade after the pole.

![Figure 4.5: MATLAB Simulation of Bode Plots](image)
Although knowing the output of the circuit is important, the change of impedance at node $V_1$ of Figure 4.1 is what we are looking for to see the change in oil quality. Since impedance, by itself, cannot be measured using Labview\textsuperscript{TM}, nodal analysis was done to find what the impedance is at node $V_1$ by knowing the input and output voltages.

The voltage at the input and output can easily be measured. By using nodal analysis at node $V_1$, the magnitude of impedance can be found. Since impedance is the voltage divided by the current and no current goes through the op-amp, the current through $Z_1$ must be the same as the current through $Z_2$. The impedance of the EIS sensor and $R_{oil}$ can then be calculated by using Equation 4.5. 2.5V was subtracted from $V_{out}$ to get rid of the DC voltage of the single power supply.

$$Z_{magV1} = -Vin = Vin_{out} - 2.5VR2 = -Vin_{out} - 2.5VR2$$

(4.5)

The phase was found by using a Fast Fourier Transform in Labview\textsuperscript{TM}. The phase along with the magnitude of the impedance can be used to find the real and imaginary parts of the impedance, shown in Equations 4.6 and 4.7. After obtaining the real and imaginary parts of the impedance, a Nyquist plot can be used to compare new and used oils. In order to test the EIS sensors Labview\textsuperscript{TM} was used to control the signal generator, measure the input and output voltages, and do frequency analysis.

$$Z_{real} = Z_{magV1} \cos \phi$$

(4.6)

$$Z_{im} = Z_{magV1} \sin \phi$$

(4.7)
Then a Labview™ routine began by controlling the Agilent 33220A Waveform Generator. A sine wave with a magnitude of 3V peak to peak was logarithmically swept for 10 seconds from .01Hz to 1500Hz. Only frequencies in the range of 1 Hz to 1k Hz were analyzed. It was found that by starting and ending out of the frequency range of interest the noise of the signal was greatly reduced. Two National Instruments (NI) USB-6008s were used to measure the input signal of the waveform generator and the output signal of the EIS signal conditioning circuit, Figure 4.1. Both NI devices were set to N Samples, with a sample rate of 10 kHz and the number of sample was set to 100,000 or 10 seconds at a rate of 10 kHz. The input and the output where then compared using a Dual Channel Spectral Measurement Block (DCSMB). This gave the magnitude and the phase of the EIS signal conditioning circuit at the output. In order to get the impedance at node $V_1$, Equation 4.5 needed to be found. This was accomplished by inverting the output signal, subtracting 2.5 Volts and then dividing by $R_2$ before comparing the input and the output signals in a DCSMB. The magnitude, real, and imaginary parts of the impedance was taken directly taken from a DCSMB. The phase was then calculated by taking the arctangent of the imaginary part divided by the real part of the impedance, shown in Equation 4.8. The Labview™ Block diagram used is shown in Figure 4.6.

$$\varphi = \arctan \frac{Z_{im}}{Z_{real}}$$ (4.8)
4.3 Water in Oil Test Set Up

Testing of the water in oil sensor was conducted using an Arduino. The Arduino supplied the power, ground and was programmed to find the change in capacitance. The signal conditioning for water in oil testing consisted of an oscillating circuit. Figure 4.7 shows the oscillator circuit used to test the water in oil sensor. The oscillator circuit outputs a square wave whose period changes as the capacitance of the water in oil sensor changes.
The Arduino counts the number of pulses for 1 second and calculates the period, $T$, using Equation 4.9. Equation 4.9 can be re-arranged to find the capacitance of the water in oil sensor ($C_{WIO}$), shown in Equation 4.10. The Arduino code was written by Dan Smith.

$$T = 2\ln 3 RC_{WIO}$$

(4.9)

$$C_{WIO} = \frac{T}{2\ln 3 R}$$

(4.10)

To test for water in oil, the Water in Oil sensor was submerged in 400 mL of virgin oil. The oil was then heated to 50$^\circ$C on a hot plate to eliminate temperature as a variable. The hotplate temperature was set to 60$^\circ$C to keep the oil at 50$^\circ$C. In order to mix the water and oil a spinner was placed at the bottom of the oil and spun during the entire test at 1500 rpm. The oil was left alone for 10 minutes to get a baseline and then .3 mL or 750 ppm of water was added every 10 minutes until there was 3 mL or 7500 ppm of water in the oil.

### 4.4 Temperature Test Set-up

Labview™ and the NI USB-6008s were again used to test the temperature sensor. In order to characterize and test the temperature sensor, they were tested in an oven while increasing and decreasing the temperature. Since the voltage of a diode changes by -2.2mV/$^\circ$C, a diode was used to calibrate the temperature sensor. A diode was used instead of a thermometer because the output voltage of a diode could be easily captured...
with Labview™, making it easy to compare the change of temperature sensor to the diode and automate the testing. Labview™ was used to measure the output voltage of the diode every 5 °C from 35 to 100°C. Figure 4.8 shows the circuit used to measure the voltage change of the diode. Table 4.1 shows the data collected from the temperature calibration.

Figure 4.8: Diode Circuit used to Calibrate Temperature Sensor

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Vout (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>35</td>
<td>0.422</td>
</tr>
<tr>
<td>40</td>
<td>0.392</td>
</tr>
<tr>
<td>45</td>
<td>0.38</td>
</tr>
<tr>
<td>50</td>
<td>0.372</td>
</tr>
<tr>
<td>55</td>
<td>0.364</td>
</tr>
<tr>
<td>60</td>
<td>0.351</td>
</tr>
<tr>
<td>65</td>
<td>0.339</td>
</tr>
<tr>
<td>70</td>
<td>0.324</td>
</tr>
<tr>
<td>75</td>
<td>0.311</td>
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<tr>
<td>80</td>
<td>0.3</td>
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<tr>
<td>85</td>
<td>0.287</td>
</tr>
<tr>
<td>90</td>
<td>0.273</td>
</tr>
<tr>
<td>95</td>
<td>0.263</td>
</tr>
<tr>
<td>100</td>
<td>0.252</td>
</tr>
</tbody>
</table>

Table 4.1: Data from the Temperature Calibration of the Diode
From this data a linear relationship was found between the output voltage of the diode and the temperature of the oven. Figure 4.9 shows the Diode Output Voltage vs. Temperature. The equation found from the best fit was, \( T = -400.59(V_{\text{diode}}) + 199.98 \), was then programmed in Labview\textsuperscript{TM} to change the voltage measured of the diode into temperature.

![Diode Output Voltage vs. Temperature](image)

**Figure 4.9: Diode Output Voltage vs. Temperature**

After calibrating the diode so temperature could be easily captured using Labview\textsuperscript{TM} the temperature sensor was ready to be tested. A half Wheatstone bridge was used with a matching resistor to set the output voltage at .3 V at room temperature for Design 1. It was difficult to consistently match the reference resistor to the temperature sensor because of variations in the thickness of the resistor. Also, since the nominal resistance was increased by ten times in Design 2 and for better repeatability, a potentiometer, which ranged from 1 to 100kΩ, replaced the reference resistor and the
output voltage was set to 0.5 volts. The circuit used to measure the temperature sensor is shown in Figure 4.10. As temperature increase the resistance of the temperature sensor increases, this changes the voltage of the half Wheatstone bridge at node V1. The voltage of the half Wheatstone bridge was then amplified by a non-inverting amplifier and recorded at the output. The gain of the non-inverting amplifier was 11V/V. The output voltage was then measured using the NI USB-6008 and the temperature was then found using Labview™.

![Figure 4.10: Temperature Test Circuit](image)

Each sensor was placed in the oven with the diode at room temperature (24°C) before the oven was turned on. The oven was then heated to 100°C and turned off. The oven was then left to cool back to room temperature with the door closed. Opening the door makes the diode cool faster than the temperature sensor. But if diode and temperature sensor are both placed in oil, the door can be opened so the oil cools quicker, this allowed for both the diode and temperature sensor to cool at similar rates. The output voltages of the diode and temperature test circuit were both measured using Labview™ and then exported to excel to be analyzed. The Labview™ block diagram used to test the temperature sensor is shown in Figure 4.11.
Figure 4.11: Labview™ Block Diagram used to Test the Temperature Sensor
Chapter 5: Testing Results and Discussion

After individual chips were packaged the individual sensors were tested using the processes described in Chapter 4. Designs 1 and 2 were both tested at RIT. Design 1’s EIS and temperature sensors were also tested by an outside company in the Fall of 2011. Design 2 will be tested by the same company at a later date. The test bench used can be seen in Figure 5.1. The test bench was able to test all devices. Beakers of clean and dirty oil were used for EIS and water in oil testing. A hotplate was used to keep the temperature constant during water in oil testing. The oven was used to test the temperature sensor. The Arduino was used for the single supply power source for all signal conditioning circuits as well as measuring the water in oil. The NI USB-6008 devices were used to capture the output voltages of the EIS and temperature outputs. A computer with Labview™ was used to capture the data from the NI devices.
5.1 EIS Test Results

5.1.1 Design 1 Testing at RIT

In order to test the EIS sensors a linear frequency was swept from 5 to 1000 Hz, instead of a log sweep, and both the input and output signals were captured. Figure 5.2 shows the Labview™ control panel used for testing Design 1. The EIS sensor on Chips 1-14 of Design 1 did not work very well. Majority of the EIS sensors were shorted together because the lines and spaces less than 20µm were not able to be fully etched. The fabrication process was then changed to account for this issue so smaller lines and spaces could be made for Design 2. Devices 15-18 worked pretty well as oil quality sensors.

![Labview Control Panel for Design 1](image)

In order to assess their functionality, Devices 15-18 were tested in the same oil to see their different responses. Then one chip was tested in three different virgin oils and
one dirty oil. The virgin oils used were Briggs Racing Oil, Royal Purple Transmission Oil, Mobil 5W-30 engine oil. The dirty oil was Briggs Racing Oil. It was important to have a clean and dirty sample of the same oil to see the difference in impedance. Figure 5.3 shows a Bode Magnitude Plot for the response of Device 18 in Mobile 5W-30 Engine Oil. As the frequency increases the magnitude decreases. This is what was expected to happen with a resistor and capacitor in parallel. This is true because as low frequencies the capacitor acts as an open and high frequencies the capacitor acts as a short, this will make the impedance be high at low frequencies and then decrease as the frequency is increased.

In order to see if the sensors have a different response to virgin and used oil, the same sensor was used to measure the same oil, virgin and used. Device 15 was used to test the difference between virgin and used Briggs Racing Oil. Figure 5.4 shows the Bode Magnitude Plot of the virgin and used Briggs Racing Oil tested by Device 15. The
used oil has a much smaller magnitude than the virgin oil. This is due to the contaminants introduced by use and wear. The magnitude should decrease, but in order to tell what made the magnitude decrease the sample would need to be sent to a lab for further testing of contaminants. But for a quick and dirty test, Device 15 showed a different response for virgin and used oil.

A Nyquist plot was then created in excel to evaluate the response of the devices between the different oils. Figure 5.5 shows the different Nyquist Plots for Device 15. As shown by the Bode Magnitude plot, the virgin and used oil each have a different Nyquist plot. This indicates that the sensor is responding to the different oils. The virgin oil has the highest resistivity and the used oil has a much smaller resistance. One problem with this data is the phase is not correct. The phase data gathered from testing Design 1 is not correct. The Nyquist plot should not curl around zero. The curl happens because Labview™ was not programmed correctly to output the phase before going into
the op-amp. The Nyquist plot still shows the difference between virgin and used oil, but does not have the correct phase.

![Nyquist Plot of Virgin and Used Briggs Racing Oil](image)

**Figure 5.5: Nyquist Plots of Device 15**

These results indicate that the fabricated EIS prototypes respond to oils with different characteristics. The designed sensors resulted in measurable impedance values, which are sensitive to the fluid being tested. More extensive testing needs to be done to determine what contaminants have the greatest impact on the impedance.

### 5.1.2 EIS Testing of Design 1 by an Outside Company

Two different EIS tests were performed by an outside company on Chips 15-18 of Design 1. The first test was done using a Garmy Reference 3000 Unit, were a frequency sweep was done to get both Bode Magnitude and Phase Plot. Each chip was placed in oil and the frequency was swept from .01Hz to 1 MHz. Figure 5.6 shows the Bode Plot of Chips 15-18. Chips 15-18 all had similar responses. The magnitude started at 1GΩ at .01Hz, but quickly fell to 100MΩ at 1Hz. The pole was around 200 Hz and the
magnitude continued to decrease as the frequency increased. The phase did exactly what was expected from 1 Hz to 1 MHz, the phase decreased to -90 degrees. This was the expected outcome that RIT tried to get from Labview™. Since the phase was correct a Nyquist Plot could be made to show the difference between the real and imaginary impedance. Figure 5.7 shows the Nyquist Plot of Chips 15-18.

Figure 5.6: Bode Plots of Chips 15-18 Tested by Outside Company

Figure 5.7: Nyquist Plot of Chips 15-18 Tested by Outside Company
The Nyquist Plot for each of the devices look very similar to what was expected for Design 1. This also shows that EIS sensors work. Each sensors Nyquist plot is slightly different because each sensor’s capacitance is different. This makes the Nyquist plot wider or shorter depending on if the capacitance is larger or smaller.

The second test, done by the outside company, was to test the devices in different samples of oil taken from the same semi-truck at different mileage intervals. The truck drove around the United States and took random samples that were analyzed by a lab to see the contaminants. Chips 15-18 were all placed in each sample the Magnitude and Phase were taken at .05Hz, .1Hz, 1Hz, 100Hz, 100kHz and 1Mhz at mileage intervals of 1876, 3701, 4929, 6235, 6674, 14429, and 17526. Each contaminant’s Impedance Magnitude vs. Contaminant in ppm were plotted to see what the contaminants had the greatest impact. Lead, Iron, Nickel, Silicon and Soot were the five contaminants that changed the most over the oil’s lifetime. They were determined to be the most important contaminants. Table 5.1 shows contaminants at the different mileage intervals.

<table>
<thead>
<tr>
<th>Mileage</th>
<th>Iron (ppm)</th>
<th>Lead (ppm)</th>
<th>Nickel (ppm)</th>
<th>Silicon (ppm)</th>
<th>Soot (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1876</td>
<td>8</td>
<td>&lt;1</td>
<td>1</td>
<td>7</td>
<td>0.2</td>
</tr>
<tr>
<td>3701</td>
<td>10</td>
<td>1</td>
<td>1</td>
<td>10</td>
<td>0.2</td>
</tr>
<tr>
<td>4929</td>
<td>10</td>
<td>1</td>
<td>2</td>
<td>11</td>
<td>0.2</td>
</tr>
<tr>
<td>6235</td>
<td>13</td>
<td>1</td>
<td>2</td>
<td>11</td>
<td>0.3</td>
</tr>
<tr>
<td>6674</td>
<td>13</td>
<td>1</td>
<td>2</td>
<td>12</td>
<td>0.3</td>
</tr>
<tr>
<td>14429</td>
<td>24</td>
<td>3</td>
<td>4</td>
<td>16</td>
<td>0.6</td>
</tr>
<tr>
<td>17526</td>
<td>33</td>
<td>5</td>
<td>7</td>
<td>17</td>
<td>0.7</td>
</tr>
</tbody>
</table>

Table 5.1: Contaminants at Different Mileages of Oil Tested
The magnitude of impedance vs. the contaminants were all plotted to see if there was a trend between contaminants. All of these contaminants had a similar plot. Figure 5.8 shows Iron’s Contaminants vs. Magnitude plot measured by Device 18. It appears that the magnitude decreases at the number of contaminants increase for frequencies less than 100Hz. It was difficult to determine what happened at high frequencies because the magnitude was much smaller than at low frequencies. The magnitude was then normalized to see the changes at each frequency. Figure 5.9 shows the Iron vs. Normalized Magnitude Measured by Device 18. The other contaminant plots can be seen in the appendix.
By normalizing the magnitude it was determined that at high frequencies it is difficult to determine what happens to the magnitude because the magnitude is already so small and becoming smaller when compared to the lower frequencies. Both graphs showed that as the number of iron contaminants increased the magnitude decreased. On this that was difficult to tell from this data is what contaminant is the most dominant? Does one contaminant dominate all the others? This data suggests that when the magnitude of the oil falls below 60% of its original magnitude the oil should be changed. In order to test this theory more oils need to be tested.

5.1.3 Design 2 Testing at RIT

Both Design A and B for EIS of Design 2 were tested by following the procedure outlined in section 4.2. All of the sensors were tested in virgin and used Valvoline Fully Synthetic 5W-30 oil. It was found that when the frequency is swept logarithmically a better response is found with less noise and there are more data points.
at the lower frequencies. The magnitude and phase were also programmed to be displayed in Labview™, but the Nyquist plots were still made in excel. Figure 5.10 shows the Labview™ Control Panel used for testing Design 2. The bode magnitude plot is showed in the upper right corner. Once again as the frequency increases the magnitude decreases. The phase is shown below it in radians. The phase is still not perfect but it does go from 0 to -90 degrees. It is unclear why it goes up before it begins to go down, but this was the best phase that was able to be captured.

![Figure 5.10: Labview™ Control Panel Used to Test Design 2](image)

The magnitude and phase were exported into excel to be further analyzed, but it is nice to see sensor working before the data is exported. The Magnitude Bode Plot of Design A can be seen in Figure 5.11. The data for Design A shows that the impedance was great for the used oil. This is the opposite of what was tested in Design 1, but is not
out of the question. If water or another contaminant with a high dielectric was in the oil, it would make sense that the used oil has a larger magnitude. But this data does show that the sensor works and can tell the difference between the oils. Another possibility for the difference is fully synthetic oil was tested. The Nyquist plot for Design a can be seen in Figure 5.12. Both the Bode Magnitude Plot as well as the Nyquist Plot for Design B were similar and can be seen in the appendix.

![Design A Bode Magnitude Plot](image)

*Figure 5.11: Design A Bode Magnitude Plot*
The Nyquist plot shows that the resistance of the used oil is $2 \times 10^9 \Omega$ and which is twice as large as the resistance of the virgin oil. But unlike in Design 1 testing the Nyquist plot now is much closer to what was expected, even though the phase was not perfect. More testing should be done by the outside company a later date to double check the performance of Design 2. Design 2 EIS sensors work. The magnitude of the impedance of the bode plot was very similar to that found in Design 1 testing by the outside company and trends the same way. The phase found was much closer to what was found by the outside company when testing Design 1, but can still be improved. The Labview™ testing works for a quick and dirty method of testing the quality of oil, but in order to know exactly what is in the oil, laboratory testing is needed.

Figure 5.12: Nyquist Plot of Design A
5.2 Water in Oil Testing

5.2.1 Design 1

Design 1 device 18 and a commercial Honeywell Relative Humidity sensor were used to test for water in oil. Both sensors were placed in 400ml of oil. After the sensors were in the oil for 2000 seconds, to get a baseline, .3ml of water was added to the oil. 750ppm of water was added at 2000, 5000, 7725, 10100, 12250, and 14500 seconds. Both, device 18 and Honeywell’s sensor’s capacitance increased. Figure 5.13 shows the water in oil sensor’s data and Figure 5.14 shows the Honeywell sensors data.

![RIT Sensor Device 18](image)

**Figure 5.13: Device 18 Water in oil Sensor**

The device 18’s capacitance increased as more water is added, but it was difficult to see what the actual change of the capacitance was due to the water. The change of capacitance is in the error of the Arduino’s counting. The error in capacitance varies by .25 pF which is half of the total change in capacitance after all the water is added. It was
shown that the IDF with polyimide can detect water in oil. The humps in the data are from the water escaping from the oil after time because the system was not closed.

The Honeywell sensor showed a greater change in capacitance with each drop of water than the water in oil sensor. The Honeywell sensor’s data looks better than the water in oil sensor, but it has the same error as the water in oil sensor. The larger change in capacitance is because the nominal capacitance is ten times larger than the water in oil sensor. A larger nominal capacitance is needed to see a greater change due to the water added to the oil using the Arduino so the change in capacitance is out of the error in the Arduino’s counting. In order to compare the change in capacitance of Device 18 and the Honeywell sensor both capacitances were normalized. Figure 5.15 compares the capacitance of Device 18 and the Honeywell sensor. The capacitance of Device 18 changed by 1.3%, this was pretty close to the theoretical change an IDF capacitor. With

![Honeywell Sensor Data](image.png)

Figure 5.14: Honeywell Sensor Data

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the same amount of water added the Honeywell sensor changed by 6%, but was slightly lower than the theoretical capacitance change. The Honeywell sensor was more responsive than Device 18. One possibility for why the Honeywell sensor was more sensitive than Device 18 was because the capacitor was a parallel plate instead of IDF. Although both sensors use polyimide, a parallel plate capacitor should be more sensitive to the change of water in oil because all of the capacitance is affected by the water being absorbed by the polyimide. An IDF structure has two capacitances, one through the polyimide and the other through the glass, but only the capacitance from the polyimide is affected by the water. Thus the IDF structure is less sensitive. With this being said a larger nominal capacitance of an IDF structure would be able to measure water in oil better.

![Percent Change Water in Oil](image)

Figure 5.15: Normalized Change in Capacitance of Device 18 and the Honeywell Sensor

5.2.2 Design 2
The goal of Design 2’s Water in oil Sensor was to increase the nominal capacitance so the change of capacitance is out of the error of the Arduino’s counting.
Design 2 doubled and tripled the nominal capacitance tested from Design 1, but the capacitance was much less than the Honeywell sensor. The water in oil Design A of Design 2A had a nominal capacitance around 90 pF and the Design 2B had a nominal capacitance around 60pF. Both designs were tested in 5W-30 Valvoline Synthetic Oil at 50°C with water content from 0 to 7000 ppm. Design A’s water in oil data is shown in Figure 5.16 and Design B’s data is shown in Figure 5.17.

![Water in Oil vs. Capacitance of Design A](image)

**Figure 5.16: Design A Water in Oil Data**
It was easier to see the capacitance change in Design A and B than it was for Device 18. Both designs capacitance increased pretty linearly when water was added. Either sensor could be used to measure water in oil. To compare the two designs to each other and the Honeywell sensor, the capacitance was normalized and the percent change was found. Figure 5.18 shows the percent change of Designs A and B.
Both Designs had a much greater percent capacitance change compared to Device 18 when 4500 ppm of water was in oil. Design A had a percent change of 3.3%, which is almost three times as sensitive as the Device 18. Design B had percent change of 2.8% which is more than twice as sensitive as Device 18. Both Design A and B were less sensitive than the Honeywell sensor when compared at 4500 ppm of water in oil. Both designs were an improvement in sensing water in oil and were out of the Arduino’s counting error.

5.3 Temperature Sensor Testing

5.3.1 Design 1 Testing at RIT
Two wafers of Design 1 were processed separately at RIT. The first wafer fabricated had beta-Ta where the second wafer had alpha-Ta. This was not good for repeatability because beta-Ta has a negative temperature coefficient of resistance of -
250ppm/°C and alpha-Ta has a positive temperature coefficient between 125 and 250 ppm/°C. It was determined that the thickness of the Ta determines the crystal structure and temperature coefficient of resistance. Thicker Ta, thicker than 2500Å, will be beta and thinner Ta will be alpha.

The first temperature sensor tested with a beta-Ta resistor was tested without Labview™ and the signal processing circuitry described in Chapter 4.4. Instead the temperature sensor was placed in oil, which was heated on a hotplate. The resistance was then taken every five minutes at temperatures ranging from 5 to 141°C. Figure 5.19 shows the change in temperature vs. resistance of the temperature sensor. The thermal coefficient of the Ta used was -250ppm/°C. A better process was implemented for the remainder of the testing, but this quick measurement showed that the Ta changed resistance as temperature changed.

![Figure 5.19: Change in Resistance with Varying Temperatures](image)
The testing procedure described in section 4.4 was used to test sensors from wafer 2 of Design 1. The Labview™ control panel for temperature testing can be seen in Figure 5.20. Both the voltage of the diode and resistor are measured and the temperature is calculated. The data was then extracted to excel to be analyzed.

Three different temperature sensors were tested from wafer 2 of Design 1 with the same design at RIT. It was found that the resistors had different resistances. The resistances varied between each device because the Ta was not uniform over the entire wafer. The thickness of Ta varies from the middle to outside because a 4 inch target was used to deposit the Ta onto a 6 inch wafer. The thickness of the Ta is thicker in the middle of the wafer than it is on the perimeter of the wafer. This means that the resistance of device from the middle will have lower resistance than the devices from the
outside of the wafer. The three temperature sensors tested had resistances of 8.6k Ω, 8.9k Ω and 10.2k Ω. The data from the test can be seen in Figure 5.21. The data shows that as the temperature increases the voltage of the output increases. This is due to the change in resistance of the temperature sensor because as the resistance increases the voltage at the Wheatstone Bridge increases. The interesting part about this data is the slopes of the different resistors don’t match even though they are the same material, Ta. It was believed that there was a constant change in resistance for Ta.

![Figure 5.21: V_out vs. Temperature of Three Different Temperature Sensors](image)

Depending on the crystal structure, Ta can either be alpha or beta. Alpha-Ta has a positive temperature coefficient where beta-Ta has a negative temperature coefficient. It was believed that the variation in thickness would only have an effect on the difference in resistance and not change the temperature coefficient. This would have resulted with data that had the same slope. Since that is not what happened, a theoretical model was made to see how what makes the slope of change. When changing the resistance of the
sensor there was no change in slope with similar the sized resistors, 5k to 15k. The actual data showed that there was a big difference between the slope of 8.6kΩ and 10.2kΩ. The next parameter that was changed was the temperature coefficient. A change in the temperature coefficients changed the slope significantly without changing from alpha-Ta to beta-Ta. Figure 5.22 shows the theoretical slopes for temperature coefficients of 125 ppm, 230 ppm and 240 ppm. The theoretical slopes match the actual slopes very well. This led to the conclusion that depending on the thickness of the Ta there are a range of temperature coefficients as well as two different crystal structures.

![Theoretical Change Vout vs. Temperature with different Temperature Coefficients](image)

**Figure 5.22: Theoretical Change in V\text{out} vs. Temperature with Different Temperature Coefficients**

To make sure that the tests are repeatable the same temperature sensor was tested five times, seen in Figure 5.23. All of the slopes are within 2% of each other and the y intercept is within 41%. The y-intercept value of test 1 was wrong because the rail voltage was not set up correctly and adjusted after test 1 for the rest of the tests. Ignoring the y-intercept of test 1 the accuracy was 4%, shown in Table 5.2. This shows that the
same resistor will produce the same voltage at a given temperature and can accurately measure the temperature.

![Graph of Vout vs. Temperature with Device 16-1 Tested Repeatedly](image)

**Figure 5.23: V\textsubscript{out} vs. Temperature with Device 16-1 Tested Repeatedly**

<table>
<thead>
<tr>
<th>Test</th>
<th>slope</th>
<th>Intercept</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>167.56</td>
<td>6.128</td>
</tr>
<tr>
<td>2</td>
<td>159.09</td>
<td>26.698</td>
</tr>
<tr>
<td>3</td>
<td>160.84</td>
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</tr>
<tr>
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<td>162.54</td>
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</tr>
<tr>
<td>5</td>
<td>162.03</td>
<td>28.492</td>
</tr>
<tr>
<td>Avg</td>
<td>162.412</td>
<td>23.7188</td>
</tr>
<tr>
<td>std</td>
<td>3.169033</td>
<td>9.883261</td>
</tr>
<tr>
<td>accuracy</td>
<td>1.95%</td>
<td>41.67%</td>
</tr>
</tbody>
</table>

| Avg (w/o 1) | 161.125 | 28.1165 |
| std (w/o 1) | 1.532286| 1.143112|
| accuracy    | 0.95%   | 4.07%   |

**Table 5.2: Shows the slopes of Device 16-1**

Temperature sensor testing showed that the uniformity of the Ta leads to a change in the temperature coefficient without changing the crystal structure. The thickness of the Ta also determines the sensitivity of the sensor. The thinner the Ta, the higher the temperature coefficient. A more uniform Ta sputter would lead to a better uniformity
throughout the wafer. The signal processing worked and the sensor repeatability was very good.

5.3.2 Design 1 Testing by Outside Company

The same three sensors that were tested at RIT were also tested by an outside company. Similar results were found by an outside company while testing Design 1’s temperature sensor testing. The outside company tested the temperature sensor by placing both the temperature sensor and a Viasala Temperature Sensor, a commercial temperature sensor, into a beaker. The temperature was swept from 10°C to 120°C by intervals of 10. The temperature sensor was then calibrated to the Viasala Temperature Environment. The calibration was similar to the calibration done at RIT with the diode. A half Wheatstone bridge was also used for signal processing and the output voltage was measured. The data gathered from the outside company can be seen in Figure 5.24.

![Figure 5.24: Temperature Testing Data from Outside Company](image)

One difference between the outside company’s testing and the testing done at RIT is that at RIT the reference resistor was changed to make sure the test started at the same output voltage. The outside company set up the test for the temperature sensor with the
resistance of 10.2kΩ and then used the same resistor for all of the testing. By changing the reference resistor all of the devices have the same starting voltage and you will never get a negative output voltage. The outside companies slope’s are very similar to that measured at RIT.

5.3.3 Design 2 Tested at RIT
The testing of Design 2 was done at RIT using the procedure described in section 4.4. The temperature sensor was tested on three different chips. The resistances again varied from sensor to sensor, but this was accounted for by changing the potentiometer so that the starting output voltage was always .5 V. Figure 5.25 shows three different temperature sensors being tested. The slope of all of the devices was very similar. Design 2 successfully was able to measure the temperature of the oven and did not have the problems associated with Design 1. Ti/Ni worked much as a better temperature sensor than Ta because the temperature coefficient of resistance was always the positive. Design 2 also worked as a better temperature sensor because its resistance was much higher than in Design 1. Having a higher resistance gives a larger output voltage making it easier to find the change in resistance. The output voltage of Design 1 changed from .3V to .8V where Design 2’s output voltage changed from .5V to 3.5V. This is due to the higher starting resistance and also the difference between the temperature coefficients of Ta vs. Ti/Ni. Ti/Ni has a much higher temperature coefficient of resistance than Ta. The temperature coefficient of resist for Design 2 was 3000ppm compared to 600ppm or -400ppm for Ta. This is less than the theoretical temperature coefficient of resist of Ni
because there is also a Ti resistor in parallel, which decreases the temperature coefficient of resist.

Figure 5.25: Design 2 Temperature Testing
Conclusion

Two revisions of the industrial fluids multi-sensor were successfully designed, fabricated and tested at RIT. The fabrication process was refined from Design 1 to Design 2 to increase the nominal capacitance of the water in oil sensor. The Ta thermal coefficient of resistance problem was solved in Design 2 by changing the metal to Ti/Ni. Design 2 successfully integrated all three sensors onto one chip. Both Design 1 and Design 2 showed working EIS, water in oil and temperature sensors. Design 2 showed major improvements in sensing water in oil and temperature from Design 1. The functionality of all sensors were able to be tested at RIT, but there is room for improvement.

A processing improvement could be made to apply the polyimide. A second mask and lithography step would be needed to spin on, develop and etch the polyimide so it is only over the water in oil sensor. This was not done for this project to save money and was not needed to prove functionality of the water in oil sensors, but would be an improvement over the post processing applying of polyimide.

One improvement to the EIS testing would be a more accurate way to test the phase at RIT. The easiest way would be to do a frequency sweep using an LCR meter. Another possibility would reprogram the Labview™ code to find the frequency and phase between the input and output signals then calculate the phase. Another possibility would be to do all testing analysis using MATLAB.
More testing should also be done by the outside company to calibrate the EIS sensor to the different contaminants and additives seen in oil through its lifetime. One way of doing this would be run EIS tests while increasing one contaminant. This way it would be easier to determine which contaminant dominates the change in magnitude. Another possibility would be to test the sensor in different oils under typical use in different trucks. This would probably show that there is a correlation between the magnitude of the impedance and when the oil should be changed. This would allow oil changes to be done based on the quality of the oil rather than the miles driven.

The next step for this project would be to package the sensor to be able to operate inside the engine. One possibility would be to use a ceramic substrate with the signal processing away from the heat of oil. High temperature epoxy would also have to be used to mount the sensor to the substrate. A flip chip would not work well for this design because the parasitic capacitance through the glass would dominate the EIS and water in oil’s capacitance making the sensors less sensitive to the oil.
## Appendix

### Process Flow for Design 2

<table>
<thead>
<tr>
<th>Step</th>
<th>Process</th>
<th>Recipes &amp; Conditions</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6&quot; Glass Wafer</td>
<td>500um Thick, 150mm Diameter</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Clean</td>
<td>Piranha Clean, H2SO4/H2O2, 130°C, 15 min., DI Rinse 5 min., SRD (Spin Rinse Dry)</td>
<td>Run Through GaSonic Asher Recipe FF</td>
</tr>
<tr>
<td>3</td>
<td>Coat with Photoresist</td>
<td>SSI Track 1 using Recipe NLOFCOTG</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>HMDS Vapor Prime, 140°C Bake, 3 min, Cool 30 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Negative Photoresist, AZ nLOF 2020, 2500rpm, 60 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Bake 110°C, 3 min</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Expose</td>
<td>ASML Stepper using NYSERDA-Fuller, Level 1</td>
<td>Dose=66mj/cm</td>
</tr>
<tr>
<td>5</td>
<td>Develop Photoresist</td>
<td>SSI Track 2 using Recipe DEVNLOFG</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>PED (Image Reversal Bake) Bake 110°C, 2 min, Cool 30 Sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Spin at 50 rpm and Dispense CD-26 for 5 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dispense CD-26 for 5 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Develop 70 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Spin at 1000 rpm and Rinse with DI water for 30 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Spin Dry at 3750 rpm for 30 sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>No Hard Bake</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Evaporate</td>
<td>CVC Evaporator- Ti Immediately Followed by Ni</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Use Ti Ni pellets</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ti Pellets- Kurt J. Lesker Materials- 99.995% Ti</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>PN#EVMTI45EXE-A</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Place 1 pellet of Ti 1st W boat, 1 Pellet of Ni in both the 2nd and 3rd W boat</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ni Pellets- Kurt J. Lesker Materials- 99.98% Ni</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>PN#EVMNI38EXEB</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pressure prior to evaporation 2E-6 Torr</td>
<td>Use W weaved baskets and make sure the pellets do</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<tr>
<td>---</td>
<td>---</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>not fall out the bottom when rotated</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>By splitting the Ni into separate baskets more Ni is evaporate and takes less power</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Increase power slowly to 220-240V for Ti</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Evaporate to Completion (look for empty boat)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Watch for empty boat</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pressure prior to evaporation 2E-6 Torr</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>.110g of Ti gives ~300Å at 25 cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>.218g of Ni gives ~300Å (2 pellets=600Å)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Evaporate 1st pellet of Ni, Increase Power Slowly to 210-220V for Ni, Evaporate to Completion</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pressure prior to evaporation 2E-6 Torr</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Evaporate 2nd pellet of Ni, Increase Power Slowly to 210-220V for Ni, Evaporate to Completion</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Lift-Off</td>
<td>Acetone and ultrasonic (Even though instructions say no Acetone)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>No heater, 1/2 inch of Acetone in a square dish, Use dirty dishes on top of cabinet</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Use Magnet to collect Ti/Ni as it lifts off, wipe Ti/Ni on clean room wipe</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rinse in DI water for 5 min, dispose of acetone safely</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>SRD</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Inspect</td>
<td>Inspect under a microscope</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Saw Wafer</td>
<td>Mount Glass Substrate on dummy wafer using wax (eg. Crystalbond 509)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Melts at 71°C, Dissolves in Acetone, Heat to bond, Reheat to Remove, Acetone to clean up</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Settings: 19 Cuts in X, 19 Cuts in Y</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Index X (horizontal) cuts 6.667 mm, Index Y (Vertical) Cuts 6.667 mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cut speed 0.2mm/sec, Height 1.5mm, Cut Depth 0.8mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Test</td>
<td>Ohmmeter to check for shorts, If open then the structure could be good unless the opens are defects</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Visual check for opens (defects)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table A.1: Process Flow used for Design 2**

74
Outside Company Testing Data of Design 1

Figure A.1: Lead vs. Magnitude Measured by Device 18

Figure A.2: Lead vs. Normalized Magnitude Measured by Device 18
Figure A.3: Nickel vs. Magnitude Measured by Device 18

Figure A.4: Nickel vs. Normalized Magnitude Measured by Device 18
Figure A.5: Silicon vs. Magnitude Measured by Device 18

Figure A.6: Silicon vs. Normalized Magnitude Measured by Device 18
Figure A.7: Soot vs. Magnitude Measured by Device 18

Figure A.7: Soot vs. Normalized Magnitude Measured by Device 18
EIS Testing of Design 2 at RIT

**Design 2B Bode Magnitude Plot**

![Design 2B Bode Magnitude Plot](image1)

*Figure A.8: Design B Bode Magnitude Plot*

**Design 2B Nyquist plot**

![Design 2B Nyquist Plot](image2)

*Figure A.9: Design B Nyquist Plot*
Bibliography


