A Multi-sensor chip for monitoring the quality of drinking water

Heidi Purrington

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A Multi-Sensor Chip for Monitoring the Quality of Drinking Water

By

Heidi M. Purrington

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

Approved by:

__________________________________________ Date ______________
Dr. Lynn F. Fuller (Thesis Advisor)

__________________________________________ Date ______________
Dale E. Ewbank (Committee Member)

__________________________________________ Date ______________
Dr. Robert E. Pearson (Program Director)

__________________________________________ Date ______________
Dr. Carl Palmer (Committee Member)

__________________________________________ Date ______________
Dr. Sohail Dianat (Chair Electrical and Microelectronic Engineering)

DEPARTMENT OF
ELECTRICAL AND MICROELECTRONIC ENGINEERING
KATE GLEASON COLLEGE OF ENGINEERING
ROCHESTER INSTITUTE OF TECHNOLOGY
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Dedication

I dedicate this thesis to my parents, Lorelei and Ross Purrington, and to my sister, Celina. There are no words to describe the amount of unconditional support and patience you have always given me. I am so fortunate to have a family who never fails to keep in mind what is truly important in life. I love you!
Acknowledgments

I would like to thank my advisor Dr. Lynn Fuller for his expertise and guidance throughout every phase of this project, and also for helping me become a more confident engineer and person. Thank you to my thesis committee members Dr. Robert Pearson, Professor Dale Ewbank, and Dr. Carl Palmer for your time, interest, and guidance. Thank you to Impact Technologies, and especially to David Ortiz, for introducing and sustaining this research opportunity. Thank you to Ivan Puchades for your help and sharp ideas. Thank you to Team Galt, my classmates, and “grad office” peers for the valuable teamwork. Thank you to Sara Widland for helping me stay on track. Thank you to the SMFL engineers and staff who keep the lab running. Thank you to my family (see dedication!). Finally, a giant THANK YOU to Murat Baylav and Chelsea Plourde for all the laughs and venting sessions and coffee breaks we shared. In you I have found lifelong friends. Thank you!
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Abstract

Drinking water should meet quality standards for the safety and satisfaction of the consumer. Quality parameters of interest include turbidity, total dissolved solids (TDS), and temperature. It is desirable to monitor these parameters in typically good sources of water to indicate if and when the water becomes unacceptable for drinking. The Environmental Protection Agency recommends that turbidity levels in drinking water remain under 5 NTU and that TDS levels remain under 500 mg/L.

A MEMS based multi-sensor chip has been developed for the purpose of monitoring turbidity, TDS, and temperature in a sample of drinking water. The benefits of MEMS technology over conventional sensors include compact size, low power consumption, integration capability, and low cost bulk manufacturing. Two revisions of the multi-sensor silicon chip were designed, fabricated, and tested. The layout of the chip and the fabrication process were redesigned in the second spin to provide more robust and sensitive responses.

The sensor structures include photodiodes, temperature diodes, interdigitated electrodes in direct contact with the sample, and capacitive interdigitated fingers. The sensors were characterized with the use of commercially available thermometers, turbidity standard solutions, and TDS standard solutions. Signal conditioning circuitry was implemented to convert each sensor output to a DC level between 0 and 1 V. The sensors were shown to be responsive to temperature, turbidity, and TDS in the ranges applicable to drinking water, although obstacles relating to reliability and signal conditioning still remain.
Chapter 1

Introduction and Motivation

It has been a growing trend in modern times to monitor and regulate potential hazards to the environment and the health of people or animals. Clean water is essential to the well being of all three aforementioned subjects. Drinking water especially should meet quality standards for both the safety and satisfaction of the consumer. This document describes the design, fabrication, and testing of a silicon multi-sensor chip that is intended to monitor a known good source of drinking water to indicate if and when it goes bad.

Two revisions of the chip were cycled through the process of design, fabrication, and test. They are referred to throughout this document as Rev. 1 and Rev. 2. Because the second revision was based on improvements to the first, the two generations of the chip are also referred to as the original design and the improved design.

The motivation for the project was to improve upon conventional methods of testing drinking water by using a micro-electro-mechanical (MEMS) based solution. MEMS sensor technology boasts many advantages over conventional sensor technology; many of which were outlined by Middelhoek et al. [1]. MEMS chips are compact in size, making them ideal for in-line testing in pipes, or other confined spaces. They are lightweight, provide low power consumption, and are accurate and robust. A variety of sensors can be fabricated into the same silicon, which provides the basis for the multi-sensor chip described in this thesis. With the appropriate fabrication process, the MEMS sensors could be integrated with on-chip CMOS signal conditioning circuitry.
Manufacturing MEMS sensor chips in bulk would also be cost-effective and would make the sensors easily replaceable.

Multi-sensor chips have been designed for various types of applications. Moreno described an “electronic tongue” that combined ion selective field effect transistors (ISFETs), interdigitated electrodes, and a silicon temperature diode on a chip to classify different brands of bottled mineral water [2]. Tang presented work towards developing a multi-sensor microsystem for measuring pH, conductivity, dissolved oxygen concentration, and temperature for industrial and biomedical applications [3].

The multi-sensor chip presented in this paper is also intended to be part of a larger microsystem. The chip will be packaged and interfaced with signal conditioning electronics, power management circuitry, and a microprocessor chip that is capable of any required real-time signal processing, temperature compensation, and data storage. A block diagram of a prototype for the envisioned microsystem is shown in Figure 1.1.

Figure 1.1 Block diagram of a microsystem interfacing with the multi-sensor chip.
The scope of this project is not to develop the entire microsystem, but to design, fabricate, and test a multi-sensor chip for monitoring water temperature, turbidity, and total dissolved solids (TDS). The structures on the silicon chip include photodiodes, interdigitated electrodes in contact with the solution, capacitive interdigitated electrodes, and a temperature diode. The individual sensors will require calibration before being used in the field, but that task is not presented here.

The multi-sensor chip is intended for an application where the source of water under test is typically good. That is, the temperature, turbidity, and TDS levels are typically within the normal expected range for drinking water. The purpose of the sensor would be to cause an alert when one or more of the elements measures beyond its acceptable range, which would prompt a more thorough investigation into the condition of the water and the root cause of the problem.

The motivation for this project is based on drinking water, but the multi-sensor chip could be used in a variety of other applications. Water quality is important for water sanitation facilities, industrial manufacturing facilities, agricultural studies, ecosystem studies, domestic applications, and recreational applications. Sensor requirements such as range and sensitivity would likely change for each different application.

The multi-sensor chips were designed, fabricated, and tested between December of 2008 and May of 2010. The chip design and layout were completed using Mentor Graphics IC software. The wafers were fabricated in the Semiconductor and Microsystems Fabrication Laboratory (SMFL) at the Rochester Institute of Technology. The sensors were characterized using laboratory equipment and purchased calibration standards.
Chapter 2

Background Information

The sensors on the chip should respond to their respective measurands in the range applicable to drinking water. The Environmental Protection Agency (EPA) has developed a set of National Primary Drinking Water Regulations, or primary standards, which are legally enforceable standards that apply to public water systems [4]. The EPA has also developed National Secondary Drinking Water Regulations, or secondary standards, which are non-enforceable guidelines regulating contaminants in drinking water that may cause cosmetic or aesthetic effects. The primary standards set a turbidity limit of 1 NTU for systems that use conventional or direct filtration. Turbidity levels for systems that do not use conventional or direct filtration must be limited to 5 NTU. The secondary standards recommend that TDS levels remain below 500 mg/L. Further background information about each parameter is provided in the following sections.

2.1 Turbidity Background

Turbidity refers to the cloudy appearance of water due to the presence of suspended particles. This could include clay, silt, sand, organic particles, or other microscopic organisms. The water appears hazy as a result of incident light scattering off of the particulate matter [5]. Turbidity can be measured optically with a turbidimeter in nephelometric turbidity units (NTU). Potential sources of interference with turbidity
measurements include air bubbles, rapid settling debris, and film build-up on the instrumentation.

The angular distribution of the scattered light depends on the wavelength, $\lambda$, of the incident light, the refractive indices of particles and test fluid, and the color, size, and shape of the suspended particles [5]. Particles that are much smaller than the wavelength of the incident light cause the light to scatter both forward and backward. Large particles cause light to scatter forward in the shape of a cone. As a result, there is a great deal of variability associated with turbidity measurements. Two samples of water with different particle compositions might produce the same turbidity reading.

Further complicating the measurement, the existing standard methods for measuring turbidity allow some flexibility in the design of turbidimeters. As a consequence, turbidimeters from different manufacturers might respond with different turbidity readings for the same water sample.

Commercially available turbidimeters may or may not comply with standard measurement methods. The EPA 180.1 method requires a tungsten-filament lamp (TFL) source, which has a peak emittance near 850 nm [5]. A silicon photodiode measures the amount of light scattered at 90° from the particles, as illustrated in Figure 2.1. This method is referred to as the nephelometric method. TFLs are rugged and inexpensive, but they are bulky and impractical for testing in situ. They also consume a lot of power and will dim over time.
The Great Lakes Instruments (GLI) method 2 is based on two orthogonal source-detector pairs [5]. The sources are infrared emitting diodes (IRED) and are turned on one at a time. The detectors measure the scattered and transmitted light intensities from each source. A microprocessor calculates the ratios of transmitted to scattered intensities to eliminate common mode effects such as fluctuating IRED power. Although this method is advantageous for eliminating sources of error, it is also much more complicated to implement. Uneven sources of interference, such as uneven window fouling, can still result in an incorrect measurement.

The two standard methods described above are for measuring low turbidity levels in the range of 0 to 40 NTU [5]. Turbidimeters are typically calibrated with the use of turbidity standard solutions. The primary turbidity standard is called formazin, a synthetic polymer solution developed in 1926 [5]. The proper way to make formazin for calibration purposes is to make a stock batch of 4000 NTU solution, and then dilute the mixture to get the desired turbidity level [6]. The reason for this is that the shelf life of formazin solution for calibration purposes decreases for lower concentrations. Formazin particles
are generally about 1 µm in size [6]. However, the micrograph in Figure 2.2 shows that formazin particles can vary greatly in size and shape, which is a good representation of the diversity of particles that might be present in any given source of water.

![Magnified image of formazin particles](image)

Figure 2.2. Magnified image of formazin particles [6].

### 2.2 Total Dissolved Solids Background

The term total dissolved solids (TDS) refers to the total concentration of ions in water contributing to electrical conduction. Ions in water come from the dissociation of acids, bases, salts, and gasses, as well as the dissociation of water itself into hydrogen (H⁺) and hydroxyl (OH⁻) [7]. The concentration of TDS can therefore be approximated by measuring the conductivity of the water sample. Pure de-ionized (DI) water is highly resistive (18.3 MΩ-cm at 25°C) and corresponds to a conductivity of 0.1 µS/cm [7]. Conductivity measurements are converted to TDS concentrations expressed in ppm or mg/L. The conversion is simple for low TDS levels. 1 µS/cm is roughly equivalent to 0.64 ppm or 0.64 mg/L [8]. The EPA recommended TDS limit is 500 mg/L [4], which would correspond to a water conductivity level of 782 µS/cm.
Conductivity measurements can only provide information about the total combined effect of all ions in the water sample. The identity and concentration of a specific ion cannot be determined by a conductivity measurement alone [7]. Monitoring TDS is further complicated at high concentrations because ion-ion interaction will introduce non-linearity into the conductivity measurement [7]. For very high TDS concentrations, the conductivity measurement will begin to level off or even decrease. Calibration and full device characterization is especially important for measurements showing non-linearity.

Common techniques for measuring the conductivity of water include direct contact or inductive type measurements [7]. Direct contact refers to measuring the conductivity of a water sample with metal electrodes that are exposed to the solution. The current flowing between the electrodes depends on the applied voltage, the TDS concentration, and the cell constant of the electrodes [7]. The major disadvantage of this technique is the potential for corrosion or coating of the sensor electrodes.

An inductive measurement requires passing voltage through a toroidal coil and measuring the current induced in another coil [7]. This technique avoids direct contact with the solution, but the coils are bulky and sensitivity is reduced. The TDS sensor on the multi-sensor chip is a direct contact conductivity sensor. The structures are metal interdigitated fingers that are exposed to the water sample.
2.3 Temperature Background

Temperature is an important parameter to measure throughout various areas of a drinking water holding tank, filtration system, purification system, etc. Temperatures out of specification could indicate a problem with the source of water or with the operation of the system. Monitoring temperature is also important because temperature compensation is usually required to maintain the accuracy of other sensors on the chip.

Common types of temperature sensors include bimetallic strips, liquid bulb thermometers, thermocouples, thermistors, and resistance-temperature detectors [9]. Each type of sensor has its own series of pros and cons. A diode is yet another type of temperature sensor, and was chosen for the design of the multi-sensor chip for its simplicity, wide linear operating range, robustness, and ease of integration in silicon.
Chapter 3

Theory and Design

Mentor Graphics IC software was used to design each revision of the multi-sensor chip. The sensors were designed with a 5 μm design rule, or $\lambda = 5 \, \mu m$. Lines and spaces were required to be a minimum of $2\lambda$. The dimensions of the Rev. 1 chip were limited to 4 mm x 4 mm due to constraints from sharing wafer real estate with other projects. The dimensions of the Rev. 2 chip were increased to 9 mm x 9 mm for a total area of 81 mm$^2$. The size increase was possible due to the flexibility of a new fabrication process customized for the multi-sensor chip. The total surface area increased from Rev. 1 to Rev. 2 by a factor of five, which allowed for larger, more sensitive devices. The two layouts, shown approximately to scale in relation to each other, are shown in Figure 3.1.

![Side-by-side comparison of the Rev. 1 and Rev. 2 chip layouts.](image-url)

Figure 3.1. Side-by-side comparison of the Rev. 1 and Rev. 2 chip layouts.
The devices on the Rev. 1 multi-sensor chip include photodiodes, a temperature diode, and three sets of metal interdigitated electrodes that are exposed to the drinking water sample. A closer look at the Rev. 1 layout is shown in Figure 3.2 with labels pointing to the various devices.

![Figure 3.2. Sensor layout for the Rev. 1 design.](image)

The turbidity photodiode is actually a large array of photodiodes connected in parallel. The device has a single n-type contact at the top of the chip and a single p-type contact at the bottom of the chip. The temperature diode is a single p-n junction with the n-type contact to the left and the p-type contact to the right. The smaller identical sets of interdigitated electrodes were intended to be sensors for TDS and free chlorine. The larger set of electrodes in the bottom right corner of the chip was intended to be a biofilm sensor. However, the free chlorine and biofilm sensors were never further developed, and the larger set of electrodes proved to be a more effective TDS sensor.
The devices on the Rev. 2 multi-sensor chip still include photodiodes, a temperature diode, and metal interdigitated electrodes that are exposed to the drinking water sample. Another set of metal interdigitated fingers, this time capacitive in nature and protected with oxide, was added to the design to serve as a water level sensor. A closer look at the Rev. 2 layout is shown in Figure 3.3 with labels pointing to the various devices.

Figure 3.3. Sensor layout for the Rev. 2 design.

The turbidity sensor is still an array of photodiodes in parallel, but the total area of the sensor is significantly larger than the Rev.1 design. The size of the photodiode was increased to allow it to capture more light scattered from the suspended particles, thus increasing the sensitivity of the device.

In the upper right corner of the chip is a single temperature diode that is identical to the temperature diode from the Rev. 1 design. It was included on the Rev. 2 design as a backup structure for temperature sensing in case the new structure did not work. The newly designed temperature sensor is located to the left of the single temperature diode.
The new design consists of five temperature diodes connected in series. The purpose of connecting the temperature diodes in series is to enhance the magnitude of the sensor response for a given change in temperature.

The design of the interdigitated electrodes for the Rev. 2 TDS sensor was based on outcomes from testing the Rev. 1 design. It was found that a large number of thin interdigitated fingers resulted in a stronger sensor response than a small number of wide fingers. The total number of fingers designed into a given area on the chip was limited by the fact that the lines and spaces must be sufficiently wide. The metal lines should be wide enough to make good contact with the water sample and to not cause reliability issues that result in open lines. The spaces must be wide enough to avoid unintentionally shorting the electrodes together with conductive particles. The final design for the Rev. 2 TDS sensor includes 150 interdigitated electrodes with 25 µm lines and 15 µm spaces. The total area of the TDS sensor was increased from Rev. 1 to further enhance the sensor response.

The smaller set of interdigitated electrodes in the bottom right corner of the chip is protected from the water sample with a layer of oxide and serves as a capacitive water level sensor. The structure was added to the Rev. 2 design to ensure that the chip is actually submerged in water when sensor data is collected. The small rectangle in the upper right corner is a p+ contact to the p-type substrate. This was added to ensure that a voltage potential could be applied to the substrate. Further details about the theoretical concepts and design of each sensor are provided in the following sections.
3.1 Turbidity Sensor Theory and Design

The turbidity sensor is a photodiode that generates current when light is scattered from suspended particles in the solution and is absorbed in the depletion region of the photodiode p-n junction. A diagram of the turbidity sensor is shown in Figure 3.4 with the LED illustrating that the incident light is initially directed parallel to the face of the photodiode before it is scattered down at an angle of approximately 90°.

![Diagram of light striking the turbidity photodiode after being scattered at 90° by a suspended particle.](image)

The structure of the turbidity sensor is a planar array of photodiodes that are connected in parallel. The parallel design is preferred over a single large area photodiode because the multiple p-n junctions translate into multiple depletion regions that capture light and generate current. This design also reduces the parasitic series resistance by shortening the distance that the current must travel between the junction and the metal contact [10]. The trade-off is that the parallel design requires more metal to make contact with each separate doped region, which blocks some of the light from reaching the substrate. The total area of the photodiode was significantly increased from the Rev. 1 design to the Rev. 2 design to enhance the magnitude of the response and the sensitivity of the turbidity sensor.
The energy of a photon is described by the well known equation

\[ E = \frac{hc}{\lambda} \]  

Equation 1

where \( h \) is Planck’s constant, \( \lambda \) is wavelength, and \( c \) is the speed of light, \( 3 \times 10^8 \) m/s. A photon will generate an electron-hole pair in the depletion region of a p-n junction if its energy is greater than the bandgap of the material. The built-in potential across the depletion region causes the newly generated carriers to be swept out of the junction as drift current [10]. The bandgap of silicon is 1.12 eV [10] at room temperature, which corresponds to wavelengths shorter than 1100 nm [10]. A typical silicon photodiode exhibits a peak response to wavelengths around 950 nm, as shown by the dotted line in Figure 3.5.

![Figure 3.5. Spectral responsivity of different types of photodiodes [10].](image)
Optical absorption as a function of depth into a silicon substrate is exponential [10]. It is related to an absorption coefficient and is described by

$$\phi(x) = \phi(0)e^{-\alpha x}$$  \hspace{1cm} \text{Equation 2}

where $\phi(0)$ is the intensity of light at the surface of the substrate, $\phi(x)$ is the intensity of the light reaching depth $x$ into the silicon, and $\alpha$ is the absorption coefficient. The absorption coefficient is wavelength dependent, so the depth that light will penetrate into the substrate is also wavelength dependent [10]. As wavelength increases, the absorption coefficient decreases and the absorption length increases. A plot of penetration depth versus wavelength is shown in Figure 3.6. The maximum depth reached by 950 nm light is approximately 5.3 μm.

![Figure 3.6. Penetration depth of light into silicon for various wavelengths [10].](image-url)
The exponential nature of absorption means that more of the light is absorbed near the surface of the silicon. Since the carriers that contribute to photo current are the ones generated in the depletion region, it is beneficial to design a shallow p-n junction. Details related to fabricating a shallow junction are discussed in Chapter 4.

Photodiodes operate under zero bias or reverse bias conditions [10]. Bias conditions were limited in the first design because all diodes were connected together through the n-type substrate. Therefore, the Rev. 1 turbidity sensor was zero biased. The inclusion of an n-well for the Rev. 2 fabrication process made it feasible to apply a reverse bias to the Rev. 2 photodiode. Reverse biasing the photodiode widens the depletion region, which makes the sensor more responsive [10].

Other benefits of reverse biasing the diode include better linearity and reduced junction capacitance [10]. The current-voltage (IV) curves in Figure 3.7 illustrate the response of a photodiode under various voltage bias conditions and light intensities.

![Figure 3.7. IV curves of a photodiode. P₀-P₂ are different light intensities [10].](image)
Turbidity measurements are sensitive to temperature due to the increase in saturation current from thermally generated carriers. The refractive index of water has also been shown to be temperature dependent [11], which affects the scattering of light in turbid water [5]. Therefore, it is important to apply temperature compensation to turbidity measurements.

3.2 TDS Sensor Theory

The TDS sensor is a set of metal interdigitated electrodes deposited over oxide and exposed to the test solution. The conductivity of a sample of water is related to its TDS concentration [8]. The electrodes monitor the conductivity of the water by measuring the current when a small AC voltage is applied to the sensor. A cross-sectional view of an interdigitated electrode structure is illustrated in Figure 3.8 where \( w \) is the metal width, \( s \) is the spacing, and \( p \) is the overlap [12].

![Figure 3.8. Diagram of interdigitated electrodes [12].](image-url)
TDS concentration trends with conductivity, but it is sometimes easier to discuss the sensing mechanism in terms of the inverse, \textit{i.e.} resistivity. In a sample with a low concentration of TDS, the conductivity of the water will be very low and the resistivity will be very high.

Because the TDS level in drinking water should be relatively low [4], it is important for the sensor to be able to measure the conductivity of highly resistive materials. To do this, the TDS sensor must have a low cell constant [13]. The cell constant, \( c_K \), of a set of planar interdigitated electrodes was defined by Smiechowski as the proportionality between the measured resistance and the specific resistance of the solution [13]. The cell constant was described by

\[
c_K = \frac{R_B}{\rho}
\]

where \( R_B \) is the measured resistance of the solution and \( \rho \) is the specific resistance of the solution.

The cell constant depends on the geometry of the interdigitated electrodes [7]. Smiechowski stated that \( c_K \) of an array of interdigitated fingers with equal lines and spaces could be calculated by

\[
c_K = \frac{2}{N \cdot L}
\]

where \( N \) is the number of fingers and \( L \) is the overlap length [13].

The Rev. 1 multi-sensor chip included two geometrically different sets of interdigitated electrodes. Both sets had equal overlap lengths, but one set had many interdigitated fingers and one set had only four fingers. Both sets were used to measure the conductivity of a TDS standard solution. According to Equation 4, it is no surprise
that the set of electrodes with a greater number of fingers was more sensitive and produced a stronger output signal than the set with only four fingers.

For this thesis project, the exact value of the cell constant is not as important as just the concept that designing for sensitivity at low TDS concentrations requires many fingers and a large overlap. The Rev. 2 TDS sensor consists of 150 fingers and an overlap length of 4000 µm. The lines and spaces are 25 µm and 15 µm, respectively. An approximation for the cell constant can be calculated by assuming that the lines and spaces are “equal enough” to use Equation 4. Under this assumption, the cell constant of the Rev. 2 TDS sensor is $3.3 \times 10^{-2}$ cm$^{-1}$.

TDS is sensitive to temperature. As temperature increases, the mobility of ions in water increases [7]. This increase in conductivity results in a higher TDS measurement. Therefore, it is important to incorporate temperature compensation when interpreting TDS measurements. The temperature coefficient is close to linear for the TDS range applicable to drinking water [7].

### 3.3 Temperature Sensor Theory

The temperature sensor is a forward biased diode. The Shockley diode equation is

$$I_D = I_S \left( e^{\frac{q V_D}{n k T}} - 1 \right)$$

Equation 5

where $I_D$ is the diode current, $I_S$ is the reverse saturation current, $q$ is the charge on an electron, $k$ is the Boltzmann constant, $V_D$ is the voltage across the diode, $T$ is absolute
temperature, and $n$ is the diode ideality factor [14]. $I_S$ can be further described by

$$I_S = AT^\beta e^{-V_{go}/nkT}.$$  

Equation 6

where $V_{go}$ is the bandgap of silicon at absolute zero, and $A$ and $\beta$ are temperature independent material dependent parameters [14].

Equations 5 and 6 indicate that for a constant diode current, the forward voltage of the diode, $V_D$, will decrease with an increase in temperature. The response is relatively linear over the temperature range applicable to drinking water sensing, so temperature results would be accurate upon sensor calibration.

The negative temperature coefficient causes the diode IV characteristic curve to shift to the left with increasing temperature. Figure 3.9 shows the IV curves for a MAX 1811 ESD diode at four different temperatures [15].

![Figure 3.9. MAX 1811 ESD-diode characterization curves showing the temperature dependence of the diode forward voltage [15].](image-url)
The sensor design for Rev. 1 consisted of a single temperature diode. The Rev. 2 design incorporates five diodes in series. The motivation for the new design was to take advantage of the cumulative effect of multiple voltage drops in series. Because a temperature change will cause a voltage shift in each diode, the sensor response will increase by a factor of five before any signal conditioning is implemented. That is, the temperature coefficient of the Rev. 2 structure would be approximately -10 mV/°C rather than -2 mV/°C.

### 3.4 Level Sensor Theory

The purpose of the level sensor is to verify that the multi-sensor chip is actually submerged in water while data about the sample is being collected. The level sensor is a set of capacitive interdigitated fingers positioned between two layers of oxide. The illustration in Figure 3.10 is a cross section of capacitive fingers with electric field lines extending above and below the plane of the fingers.

![Figure 3.10. Cross-section illustration of the water level sensor.](image)
The level sensor exploits the difference in dielectric properties between water and air. The oxide layer deposited over the metal capacitive electrodes is designed to be sufficiently thin so that the ambient material, whether it is air or water, falls within the electric field lines and therefore has an effect on the capacitance of the sensor. The relative permittivity of air is approximately 1. The relative permittivity of water is around 80, depending on the temperature [16]. The difference in permittivity between water and air is substantial enough to cause a measurable increase in capacitance when the sensor is submerged in water compared to when water is absent. Signal processing circuitry is used to convert the capacitance value to a voltage output.

It is complicated to calculate the capacitance of a set of interdigitated electrodes. Engan first presented a model that required calculating complete elliptic integrals of the first kind [17]. His assumptions were that piezoelectric coupling can be neglected, boundary effects can be ignored, and the metal film thickness is negligible compared to the other dimensions of the structure. Otter then developed an approximate model that uses a Bessel function available in many desktop software packages such as Excel [12]. The model developed by Otter is

\[
C = pq \frac{4}{\pi a} \varepsilon \sum_{n=1}^{\infty} \frac{1}{2n-1} J_0^2 \left( \frac{(2n-1)\pi s}{2a} \right)
\]

Equation 7

where \( p \) is the overlap length, \( n \) is the number of fingers, \( s \) is the spacing, and \( a \) is the periodicity of the interdigitated electrodes. The model is further complicated for the case of the water level sensor because of the different material layers involved, but an approximation would predict the capacitance to fall in the vicinity of tens of picoFarads.
Chapter 4

Sensor Fabrication

Both revisions of the multi-sensor chip were fabricated in the SMFL at RIT. The first revision was fabricated along with other MEMS projects on 100 mm wafers with a sixty-one step MEMS process flow. One of the completed wafers is shown in Figure 4.1 with arrows pointing to a few of the drinking water quality multi-sensors. Each wafer contained about a dozen of each design.

Figure 4.1. 100 mm wafer with arrows indicating the Rev. 1 multi-sensor chips.

The Rev. 1 process flow began with n-type wafers that were thinned down to approximately 350 µm. Seven photolithography levels were completed with manual alignment. Doped p+ and n+ regions were formed through predeposition and diffusion. Aluminum was sputtered for the metal layer. The process was successfully completed after five weeks of full time processing by a team of people.
One advantage of using the RIT BULK MEMS process was that it had already been established as a robust process that would produce functional devices. Another advantage was the variety of devices that it was able to produce. There were, however, limitations associated with the process that could only be resolved by completely redesigning the flow. Therefore, as a major improvement to the second spin of the project, a new process flow was developed that included those and only those fabrication steps beneficial for the drinking water multi-sensor chip.

The Rev. 2 chips were fabricated on 150 mm (six inch) wafers, which immediately improved the process by enabling the use of some automated equipment that is not compatible with 100 mm wafers. For example, the automatic alignment of the Canon stepper tool (150 mm wafers only) performs alignment and resolution at 1 µm or better. Each 100 mm wafer contains about three dozen of each multi-sensor chip. A completed Rev. 2 wafer is shown in Figure 4.2.

![Fabricated 150 mm Rev. 2 wafer.](image)
The Rev. 2 process flow was designed to make the desired structures in a minimum number of steps to save time and money. The process began with lightly doped p-type wafers (40 Ω-cm). N-type phosphorus doped wells were developed to allow for device isolation and series diode connections. A boron channel stop implant was used in the regions outside the n-wells to prevent unwanted channel formation in the substrate. Diodes were formed by implanting shallow boron doped p+ regions inside the n-wells. Chromium was sputtered for the metal layer. Field oxide was thermally grown, and passivation oxide was deposited by low-temperature oxide (LTO). The oxide thicknesses were designed to serve as an anti-reflective coating (ARC). The purpose of each step is described more extensively in sections to follow. A detailed process flow is provided in Appendix A.

A major processing technique improvement for Rev. 2 was switching to ion implantation as the method of doping. Ion implantation is more predictable and well controlled than predeposition/diffusion. It can also create shallower junctions than predeposition/diffusion, which is beneficial for the photodiode. The boron channel-stop implant was also new to the Rev. 2 process, and was necessitated by the new sensor design and use of n-wells. The n-well mask was re-used for the channel-stop photolithography step by incorporating negative photoresist. The thirty-two step process required seven photolithography steps and was completed by one person in approximately five weeks.
4.1 Turbidity Sensor Fabrication

The Rev. 1 turbidity sensor photodiode was fabricated by diffusing doped p+ regions into an n-type substrate. The doping concentrations and junction depth were predetermined by the established RIT MEMS process flow. The junction depth was about 3 µm. The relatively deep junction depth did not make efficient use of the fact that most of the incident light is absorbed close to the surface of the substrate due to the exponential absorption characteristic.

The fabrication process for Rev. 2 was therefore improved by using ion implantation to produce shallow junctions. The shallow p+ junction was made by implanting a dose of boron $1.8 \times 10^{13}$ cm$^{-2}$ into the n-well regions at 45 keV through approximately 1000 Å of oxide. The implant was annealed for 44 minutes at 1000°C. The target junction depth was approximately 1 µm. Thermal oxide (approximately 2500 Å) was grown during the anneal step, which consumes silicon and keeps the junction closer to the surface.

The Rev. 2 p+ region was implanted into an n-well, rather than directly into the substrate. The n-well was designed to be sufficiently deep to allow for a widened depletion region with the photodiode reverse-biased. The n-well/p-substrate junction was designed to be 4 µm deep. The n-well was made by implanting a dose of phosphorus $9 \times 10^{12}$ cm$^{-2}$ into the p-type substrate at 120 keV through 1000 Å of oxide. The well drive was 24 hours at 1100°C to place the junction approximately 4 µm deep in the silicon.

The Rev. 2 fabrication process was simulated using Silvaco’s Athena software. The purpose of the simulation was not to provide an exceptionally accurate process
model, but rather to provide an approximate representation to rule out potential flaws in the design of the process flow. Mainly default settings were used throughout the simulation.

Figure 4.3 shows the simulated concentration profile of the photodiode. The starting p-type wafer resistivity was measured beforehand using the 4-point-probe technique to make the simulations more accurate. The resistivity was measured to be 40 Ω-cm, which corresponds to a boron concentration of $3 \times 10^{14}$ cm$^{-3}$. The n-well/p-substrate junction depth was simulated to be approximately 4 μm deep with a peak phosphorus concentration of $10^{15}$ cm$^{-3}$ in the n-well region. The p+/n-well junction depth was simulated to be approximately 1 μm from the silicon/oxide interface with a peak boron concentration of $10^{17}$ cm$^{-3}$ in the p+ region. The relatively high sheet resistance of this p+ region is not a problem because the photodiode operates under zero or reverse bias conditions, where the device functionality is not limited by the parasitic resistance.

Figure 4.3. Simulated diode concentration profile using the Rev. 2 process flow.
Finally, the Rev. 2 turbidity sensor was improved through fabrication methods by designing the thickness of the passivation oxide to serve as an anti-reflective coating (ARC). The oxide that separates the photodiode active silicon from the atmosphere is constructed throughout several steps in the process. First, 1000 Å are grown in 16 minutes at the very end of the 1100°C well drive. Next, 2500 Å are grown during the implant anneal for a total of 3500 Å. Finally, low-temperature oxide (LTO) is deposited after the chrome sputter as a passivation layer between the metal and the atmosphere. The total oxide thickness can be designed as an ARC to help reduce the substrate reflectivity and increase the response of the turbidity sensor.

There are two ways that an ARC can be optimized to reduce substrate reflections. The first is to choose a material with an index of refraction defined by

\[ n_{ARC} = \sqrt{n_1n_2} \]  

Equation 8

where \( n_1 \) is the refractive index of the ambient media (i.e. water), and \( n_2 \) is the refractive index of the substrate (i.e. silicon) [18]. This is an ideal condition that cannot be met with the Rev. 2 process flow. The refractive index of the ARC is constrained to that of silicon dioxide.

The other optimization technique is to choose the ARC thickness so that destructive interference occurs between the reflected components of the ARC and the substrate [18]. The ideal thickness depends on the ARC material (oxide) and the wavelength of the incident light. The criteria is described by

\[ t_{ARC} = m\frac{\lambda}{4n_{ARC}} \]  

Equation 9
where $t_{ARC}$ is the thickness of the ARC, $m$ is an odd-numbered multiple, $\lambda$ is the wavelength of the turbidity sensor LED (950 nm), and $n_{ARC}$ is the refractive index of the ARC material, SiO$_2$ (1.455) [18].

The effectiveness of the ARC depends on the thickness of the ARC and the materials in the stack. In the case of the turbidity sensor the reflections cannot be completely eliminated because of the constraint on the refractive index of the oxide.

PROLITH software was used to simulate the effects of designing the oxide over the turbidity sensor oxide as an ARC. The swing curve in Figure 4.4 shows the substrate reflectivity versus $t_{ARC}$ for an incident wavelength of 950 nm and a material stack of water over oxide over silicon. The plot shows that reflectivity maximums occur at even-quarter wavelength intervals, while reflectivity minimums occur at odd-quarter wavelength intervals. The simulation also shows that the maximum substrate reflectivity is 24.6% and could potentially be reduced to a minimum of 16.6%. The Rev. 2 process was designed such that the oxide thickness over the turbidity sensor would be 1440 nm.

![Figure 4.4. Reflectivity minima and maxima for various ARC layer thicknesses.](image-url)
4.2 TDS Sensor Fabrication

The TDS sensor is composed of metal interdigitated fingers that are exposed to the water sample and serve as electrodes to conduct current. The response of the sensor is mostly determined by the geometry and physical dimensions of the interdigitated fingers. However, there was a problem with the Rev. 1 TDS sensor due to the metal chosen for the fabrication process. Aluminum was used as the metal layer in the RIT MEMS process. Aluminum is a relatively soft and grainy metal and is susceptible to corrosion and electromigration [19].

Electromigration is a reliability issue where the grains of metal can begin to move and flow when an electric field exists between the electrodes [20]. The higher the electric field, the worse the effects can be. In the case of the Rev. 1 sensor, the presence of water appeared to accelerate the electromigration. During the testing process the TDS sensor began to fail due to low resistance between the electrodes. Inspection with a microscope showed that grains of aluminum were bridging between the metal fingers in some areas. Even if the bridging effect due to electromigration did not completely short out the device, any decrease in resistance between the electrodes resulted in decreased sensitivity to TDS. Methods for reducing the problem include applying a smaller voltage to the electrodes, widening the spaces between the fingers, and choosing a different metal for the Rev. 2 fabrication process.

The other problem with aluminum is its susceptibility to corrosion. Ionized particles in a solution can react with the metal and cause it to corrode [21]. Corrosion of the TDS electrodes will result in metal traces that are open and will not conduct.
electricity, thus disabling the sensor. Figure 4.5 (a) is a micrograph of a Rev. 1 TDS sensor that was not exposed to an electrolytic solution and has fully intact aluminum fingers. Figure 4.5 (b) is a micrograph of a sensor that was exposed to TDS conductivity standard solutions for a couple of days and exhibits extensive corrosion. The solution to this problem would be to use a metal that is less susceptible to corrosion.

Chromium was the metal chosen to resolve the electromigration and corrosion issues. Upon completed fabrication, the Rev. 2 TDS sensor was tested in the same environment and under similar conditions as the Rev. 1 sensor. After several days of exposure to the conductivity standard solutions, the chromium electrodes showed no signs of electromigration or corrosion as shown in Figure 4.5 (c).

![Figure 4.5](image)

Figure 4.5. (a) Rev. 1 intact aluminum electrodes (b) Rev. 1 corroded aluminum electrodes (c) Rev. 2 intact chromium electrodes after exposure to solution.
Chromium was deposited on the wafers using a DC sputter technique. The six-wafer lot was split at the chrome sputter step in case any misprocessing occurred. Chrome was sputtered on only two wafers at first. After the metal photolithography step, the metal was etched with a wet chemical chrome etchant. The two wafers were then placed in the Branson Asher for photoresist removal. A reaction occurred while the wafers were in the asher that caused a white film with crystalline patterns to develop over the chrome. It is likely that some type of metal oxidation occurred. Approximately 90% of the wafer had to be scrapped because the chrome had become highly resistive. A photograph of the misprocessed wafer is shown in Figure 4.6. Photoresist removal after chrome etch was done by PRS-2000 solvent strip on subsequently processed wafers.

**Post-ash film**

![Post-ash film](image)

Figure 4.6. Photograph of a white crystalline film that formed over the chrome.
4.3 Temperature Sensor Fabrication

The original temperature sensor design was a single p-n junction diode. The diode was formed by p-type diffusion into an n-type substrate. Diodes could not be connected in series because all of the n-type regions were connected together through the substrate. Furthermore, the n-type region of the temperature diode was connected to the n-type region of the photodiode, which further restricted the design of the signal conditioning circuitry.

The fabrication process was improved for the Rev. 2 design with the addition of the n-wells to allow for building diodes in series. A channel-stop implant was included in the regions surrounding the n-well so that the voltage potential on the nodes between series diodes did not induce a channel in the p-type substrate.

4.4 Level Sensor Fabrication

The water level sensor was not included on the Rev. 1 chip design, but was introduced on the Rev. 2 chip. The only major consideration for the fabrication of the levels sensor was that the oxide covering the electrodes remained waterproof. Otherwise, a drift in the capacitance might be observed as water particles absorbed into the oxide. A high quality passivation oxide would therefore be beneficial for the sensor. To this end, LTO oxide was deposited for the passivation layer rather than the lower quality TEOS.
Chapter 5

Packaging and Signal Conditioning

A certain amount of chip testing was accomplished at wafer level using a microscope and fine tipped wafer probes to touch down on the pads of the chip under test. This was a useful technique for initial testing of the devices. The wafer probes were interfaced to a computer with ICS software. I-V characteristic curves were plotted to ensure that the diodes were truly functioning as diodes. Wafer level testing was also used to verify that the photodiode was sensitive to light, and that the temperature diode was sensitive to heat.

Wafer level testing was not sufficient for fully characterizing the sensors because the application of the multi-sensor chip is in water, and it is not practical to submerge a wafer in liquid for testing purposes. Therefore, the wafers were sawed into individual chips, and then the chips were packaged onto printed circuit boards (PCBs) for more extensive testing.

PCBs were designed for the project using ExpressPCB software. The board layout was printed onto a transparency for contact lithography, and then the PCB was processed in the SMFL. The chip was secured to the board with epoxy, and then a wire bonding tool was used to connect the sensor devices to the copper traces of the PCB. Other components on the chip PCB include an infrared LED for the turbidity sensor and connector pins to interface with power supplies and signal conditioning circuitry. Finally, a water-proof coating such as nail polish or epoxy was applied to the copper traces, the
leads of the LED, the wire bonds, and any bond pads of the chip that could not be exposed to water.

The Rev. 1 and Rev. 2 chip PCB designs are shown in Figure 5.1 (a) and (b), respectively. A major improvement to the Rev. 2 PCB design was to increase the length of the copper traces from the chip to the connector pins. The chip was located too close to the connector pins in the Rev. 1 design. It was difficult to fully submerge the chip without causing problems by inadvertently wetting the connector pins.

Figure 5.1. PCB layouts for packaging the Rev. 1 (a) and Rev. 2 (b) sensor chips.

Before a signal conditioning PCB was fabricated, the circuitry was first built in a breadboard (Figure 5.2). The advantages of using a breadboard include design flexibility and the capability for fast circuit modifications. The disadvantages include noisier signals and messy wires that sometimes pop out of the board and are confusing to debug.

Figure 5.2. Breadboard implementation of the signal conditioning circuitry.
Aside from cleaner, neater signals, a PCB version of the signal processing circuitry is desired because the final application of the project will require a compact and robust way to interface with the sensor chip. Figure 5.3 shows the Rev. 1 signal conditioning PCB layout and also the fully populated board. Separate PCBs were built for the sensor chip and the signal conditioning circuitry so that different chips could be easily exchanged in and out of the system.

![Figure 5.3](image1)

(a) Rev. 1 PCB design and (b) Fabricated and populated PCB.

The placement and characteristics of the infrared LED have a great deal of impact on the performance of the turbidity sensor. Because the turbidity level of drinking water should remain below 5 NTU [4], the photodiode must be sensitive to very small concentrations of suspended particles by detecting very small changes in the amount of scattered light. Stray light reaching the photodiode directly from the LED would have a flooding effect on the sensor and would decrease the sensitivity at the smallest levels of scattering. Therefore, the infrared LED should have a narrow viewing angle, represented by $\theta$ in Figure 5.4.
Figure 5.4. The viewing angle of the light source, $\theta$, should be narrow to minimize stray light reaching the turbidity sensor.

Stability of the IR light source is a concern because fluctuations in intensity could appear as fluctuations in turbidity. It is important that the IR LED does not bend closer to or further away from the photodiode. The IR LED used for the Rev. 1 turbidity sensor was a very common type of LED package, shown in Figure 5.5 (a). The leads of the diode had to be bent for the light to shine parallel to the face of the sensor, which introduced stability problems. Furthermore, the wide viewing angle required black heat shrink to block stray light from flooding the photodiode. The improvement for Rev. 2 was to use an infrared LED with a narrow viewing angle and a “side-looking” package, as shown in Figure 5.5 (b), to make the sensor more robust by eliminating bent leads. The SFH 4110 was chosen for its side-looking package and its $\pm 9^\circ$ viewing angle.

Figure 5.5. Two LED packages: (a) traditional and (b) side-looking.
Once the multi-sensor chip was properly packaged, it was then connected to the required signal conditioning circuitry and submerged in water samples for further testing. The following sections describe the unique signal conditioning circuitry for each sensor. The circuitry was designed for ±3.3 V power supplies.

### 5.1 Turbidity Sensor Signal Conditioning

The turbidity sensor is a photodiode that generates a magnitude of current proportional to the amount of light scattered from suspended particles in the water sample. The signal conditioning circuitry for the turbidity sensor converts the photodiode current to a voltage output between 0 and 1 V (Figure 5.6).

![Figure 5.6. Signal conditioning circuitry for the turbidity sensor.](image)

The anode of the photodiode is connected to virtual ground through the inverting input of an op-amp. The cathode is connected to the positive 3.3 V supply rail, effectively reverse biasing the photodiode to enlarge the depletion region and generate a larger response. The first op-amp stage provides gain while converting photodiode current to voltage. The second op-amp stage provides more gain while incorporating a DC offset to
subtract the effects of stray light reaching the photodiode directly from the LED. The infrared light source is powered by a DC signal through a 120 Ω current limiting series resistor.

5.2 TDS Sensor Signal Conditioning

The TDS sensor is a set of electrodes that are exposed to the water sample and conduct current in proportion to the concentration of ions in the solution. The signal conditioning circuitry for the TDS sensor converts the current to a voltage output between 0 and 1 V (Figure 5.7).

![Figure 5.7. Signal conditioning circuitry for the TDS sensor.](image)

The input to the sensor is a 4 kHz sine wave. A small AC signal was used to reduce the polarization tendency of the ions by constantly changing the direction of the field. A buffer stage separates the voltage divider from the input electrode. The op-amp connected to the TDS output electrode converts the current to voltage while providing gain. The final stage is a peak detector that converts the amplitude of the output sine wave to a DC voltage level.
5.3 Temperature Sensor Signal Conditioning

The temperature sensor consists of a diode or series of diodes that exhibit a decrease in forward voltage, \( V_F \), with an increase in temperature. The Rev. 1 temperature sensor was a single diode with a forward voltage around 0.6 V at room temperature. Very little circuitry was required to convert the output to the standard 0 to 1 V DC level. The circuit with a resistor in series with the diode and power supply is shown in Figure 5.8.

![Figure 5.8. Rev. 1 simple circuit for testing the temperature diode.](image)

In an effort to improve the temperature sensor for Rev. 2, five diodes were connected in series to create a larger voltage drop per increase in temperature. This greatly affected what was required for signal conditioning circuitry because a typical output voltage for five diodes in series is up to 3 V, which does not meet the desired criteria for an output voltage between 0 and 1 V. The signal conditioning circuitry used for the Rev. 2 temperature sensor is shown in Figure 5.9.
The first op-amp stage of the circuit is a voltage buffer that is connected to the anode, or the p-type end of the series of diodes. The second amplifier stage provides gain while incorporating a DC offset to condition the output of the circuit between 0 and 1 V.

### 5.4 Level Sensor Signal Conditioning

The water level sensor is a capacitive set of metal interdigitated fingers. The capacitance increases when the chip is submerged in water compared to when water is absent. The signal conditioning circuitry converts the capacitance to a voltage output. The signal conditioning circuitry is shown in Figure 5.10.

![Figure 5.10. Signal conditioning circuitry for the water level sensor.](image)
A 0 to 3.3 V square wave at a frequency of 4 kHz is applied to the capacitive sensor through a 1 MΩ resistor. The resistor and capacitive sensor form an RC integrator circuit. With a 50% duty cycle, the input voltage is high for 125 µs. A high resistor value (1 MΩ) was chosen to ensure that the time constant, \( \tau \), of the RC integrator was longer than 125 µs. With a sufficiently long \( \tau \), the RC integrator does not have enough time to fully charge, thus the maximum voltage across the capacitor will be a fraction of the 3.3 V input. A buffer stage is added at the output of the RC circuit. A peak detector is then used to convert the maximum voltage across the capacitor to a DC level.

The increase in permittivity from air to water means that the capacitance of the sensor is higher when it is submerged in water compared to when it is not. A higher capacitance translates to a longer \( \tau \), so it will take longer to charge the capacitor. As a result, the maximum voltage across the capacitive sensor when it is submerged in water will be less than when it is not. A comparator circuit takes advantage of this effect. The reference level of the comparator must be set to a voltage that falls between the voltage levels seen on the capacitor in water and in air. The comparator is inverting, so when the sensor is in air, the max voltage across the capacitor is higher than the reference level and the comparator output is low. When the sensor is in water, the capacitor voltage is lower than the reference level and the comparator output is high, or 3.3 V. The final stage of the signal conditioning circuitry is a voltage divider to provide a final DC output of 0 or 1 V.
Chapter 6

Test Results and Discussion

A limited amount of testing and verification was initially performed on some of the devices at wafer level. Most of the testing, however, was performed on chips that were packaged on printed circuit boards and submerged in water samples. The test setup used extensively for Rev. 2 sensor verification is shown in Figure 6.1.

![Test setup for verifying each of the sensors.](image)

The test setup includes a packaged sensor chip submerged in a water sample, signal conditioning circuitry, power supplies, and an oscilloscope for signal analysis. A “dark chamber” was used to block the ambient room light from interfering with turbidity
measurements. If the sample needed to be heated to validate the temperature sensor, or to examine the effects of temperature on the other sensors, a beaker containing the sample was heated on a hot plate with a commercial thermometer to indicate the actual temperature (Figure 6.2).

![Commercial thermometer and Sensor chip](image)

*Figure 6.2. Multi-sensor chip submerged in a sample of water with a commercial temperature sensor.*

A set of turbidity standard solutions ranging from 1 NTU to 1000 NTU were ordered for the purposes of characterizing the turbidity sensor. A set of TDS standard solutions ranging from 70 to 7000 µS/cm were ordered for characterizing the TDS sensor. The following sections summarize both the Rev. 1 and Rev. 2 test results.
6.1 Turbidity Sensor Test Results

The results from testing the Rev. 1 turbidity sensor are shown in Figure 6.3. The time scale on the oscilloscope was dramatically increased so that the sensor could be submerged in turbidity samples ranging from 1 NTU to 1000 NTU, and the output voltage levels could all be viewed on the same screen shot.

Figure 6.3. Rev. 1 turbidity sensor output voltage plotted versus time while submerged in water samples of varying turbidity levels.

The screen shot shows that the output voltage increased from 360 mV in a 10 NTU sample to 820 mV in a 1000 NTU sample. The outlier is the 1 NTU sample, which does not follow the expected trend. Because 1 NTU consists of a very low concentration of particles, it may have been contaminated to a level greater than 1 NTU, although care was taken to minimize contamination by rinsing the sensor in DI water when changing from a sample of higher turbidity to lower turbidity. The sensor might
just not have been sensitive enough to provide accurate measurements at low turbidity levels. Actions were taken to improve the sensitivity for Rev. 2.

Initial testing of the Rev. 2 turbidity sensor involved verifying that the structure functioned properly as a photodiode. ICS sweeper software was used to capture the diode IV characteristic curve, shown in Figure 6.4. The plot confirms diode-like behavior of the structure; the photodiode is off during zero bias or reverse bias conditions, and is on when a sufficient forward bias voltage is applied.

An ideal forward biased diode would show an exponential IV relationship. However, the characteristic curve of the photodiode in the forward biased region is obviously linear. This indicates that the series resistance limits the diode performance under forward biased conditions. A best-fit line through the linear data indicates a slope of 2.1745 mA/V. The resistance is the inverse of the slope and was calculated to be 460 Ω. The series resistance is not a terrible concern for the photodiode because it will not be operated under forward bias conditions.

![Photodiode I-V Characteristics](image)

Figure 6.4. Photodiode I-V curve showing a series resistance of 460 Ω.
Once the diode behavior was confirmed, it was important to ensure that the photodiode actually would respond to light. The ICS sweeper software was used to apply a reverse bias to the photodiode down to -3.3 V. The chip was placed on the stage of a microscope and the intensity of the microscope lamp (white light) was varied in brightness. The plot in Figure 6.5 shows that the magnitude of the sensor response increases with brighter light, as expected. The plot also shows that the magnitude of the sensor response increases with a larger reverse bias, which is also expected. The lowest brightness level shown on the plot corresponds to the microscope being turned completely off. However, a small amount of ambient room light was still present during the test. To get a true measurement of the photodiode reverse leakage current, the chip must be placed in complete darkness.

![Photodiode Current vs. Brightness](image)

Figure 6.5. Plot of photodiode current increasing in magnitude as a larger reverse bias voltage is applied to the diode.
Initial testing verified that the structure indeed behaved like a photodiode. The next step was to package a chip on a PCB and apply signal conditioning circuitry to test the photodiode as a turbidity sensor. The results from testing the Rev. 2 sensor in standard solutions ranging from 10 NTU to 1000 NTU are shown in Figure 6.6 (a). The output voltage ranged from 14 mV to 900 mV. Figure 6.6 (b) provides a closer look at the lower turbidity concentrations.

![Turbidity Results](image)

(a)

![Zoom](image)

(b)

Figure 6.6. (a) Rev. 2 turbidity sensor output plotted versus concentration. (b) Zoomed in view of Rev. 2 turbidity results (low concentrations).
Generally, the output voltage versus turbidity level was shown to be a linear relationship. Unfortunately, the results when testing the 1 NTU sample were again inconclusive. Despite choosing an infrared LED with a narrow viewing angle, there still was an issue with flooding the photodiode with stray light. The signal conditioning circuitry was designed with an input offset to account for this factor. However, the extensive amount of gain in the circuit amplifies the noise in the DC offset, which makes the output appear unstable when trying to measure the smallest lowest levels of turbidity.

The method of reverse biasing the photodiode to capture more light may also have backfired. The intrinsic reverse current, or dark current of the photodiode increases in reverse bias mode, which may have interfered with detecting the low turbidity levels. Future work on this project might include the use of a laser diode infrared source, a more precise way to mount the source to eliminate stray light, and a return to zero bias photodiode operation.

A beaker containing a 100 NTU turbidity sample was heated on a hot plate to examine the effects of temperature on the turbidity sensor output. A commercial thermometer was included in the test setup to indicate the temperature of the sample. The results in Figure 6.7 show that there is an exponential relationship between the turbidity measurement and water temperature. This characteristic is dominated by the fact that the sensor is a photodiode and there is an exponential relationship between diode current and temperature. Temperature compensation would be required for this sensor in the application, which is one reason why a temperature sensor is included on the multi-sensor chip.
Figure 6.7. The turbidity sensor output is exponentially related to temperature.

### 6.2 TDS Sensor Test Results

The results from testing the Rev. 1 TDS sensor are shown in Figure 6.8. Again, the time scale on the oscilloscope was increased in order to view the sensor output voltage for TDS samples ranging from DI water (nearly 0 µS/cm) to 7000 µS/cm. The screen shot shows that the output voltage increased from 120 mV in DI water to 1.0 V in a 7000 µS/cm sample. The results followed the expected trend. A higher concentration of TDS caused the water sample to be more conductive. The sensor output begins at 0 V in air, which verifies that the electrodes are open and there is no conduction. The output level for the DI water does not return exactly to 120 mV because rinsing the sensor in the DI water contaminated it and raised its TDS concentration.
Figure 6.8. Rev. 1 TDS sensor output for varying TDS concentrations.

The results from testing the Rev. 2 sensor in TDS standard solutions ranging from 0 ppm to 960 ppm are shown in Figure 6.9 (a). The output voltage ranged from 7.2 mV to 945 mV. Figure 6.9 (b) provides a closer look at the lower TDS concentrations.

![Figure 6.8](image1)

![Figure 6.9](image2)

Figure 6.9. (a) Rev. 2 TDS sensor output and (b) lower TDS concentrations.
The square data points in Figure 6.9 represent the DC output of the circuit described in Section 5.2. The diamond data points represent the voltage amplitude at the output of the op-amp. The difference is due to the voltage drop across the diode in the peak detector circuit, which is slow to turn on at low TDS concentrations. At high TDS concentrations, the output of the circuit begins to level off. Clearly, this is not a straightforward linear relationship. The response needs to be fully characterized and calibrated in order to properly relate an output voltage with a TDS concentration.

A beaker containing a 70 µS/cm TDS sample was heated on a hot plate to examine the effects of temperature on the TDS sensor output. The results in Figure 6.10 show that there is an approximately linear relationship between the TDS measurement and temperature. Temperature compensation would be required for this sensor in the application.

Figure 6.10. The TDS sensor output varies linearly with temperature.
6.3 Temperature Sensor Test Results

The Rev. 1 temperature sensor was characterized using the very simple test circuit consisting of a series resistor between the temperature diode and the 3.3 V power supply. The chip was submerged in a water sample along with a commercial thermometer to indicate the temperature of the water. The forward voltage of the temperature diode is plotted versus temperature in Figure 6.11. The sensor response is linear over the temperature range applicable to drinking water, and the slope of the line shows that the temperature coefficient is -1.8 mV/°C.

![Diode Voltage vs. Temperature](image)

Figure 6.11. Rev. 1 temperature sensor results. Slope is -1.8 mV/°C.

The temperature sensor was improved for Rev. 2 by connecting five diodes in series and monitoring the voltage drop of the entire structure. The motivation was to increase sensitivity by producing a response to temperature that was five times stronger than the Rev. 1 response. More complex signal conditioning circuitry was added to the
Rev. 2 design. The circuitry incorporates gain and DC offset to utilize more of the 0 to 1 V range than the Rev. 1 sensor.

Initial testing of the Rev. 2 temperature sensor included verifying that the forward voltage drop of the diode structure changed with temperature. ICS sweeper software was used to capture the diode I-V characteristic curves (Figure 6.12). A heat gun was used to increase the temperature of the chip from room temperature to approximately 100°C. The plot confirms that the forward voltage of the diode decreases with increasing temperature, causing the I-V curve to shift to the left.

![Rev. 2 Temperature Diode I-V](image-url)

**Figure 6.12.** Characteristic curve of the Rev. 2 temperature sensor shifting to the left with increasing temperature.

The curves in Figure 6.12 were plotted at temperatures ranging from 25°C to 97°C ($\Delta T = 72°C$). At a bias current of approximately 40 $\mu$A the forward voltage of the temperature sensor shifted from 1.3 V to 0.6 V ($\Delta V_F = 700$ mV). The total sensor
response was therefore shown to be -9.7 mV/°C before any signal conditioning circuitry was applied. The contribution from each of the five individual series diodes was approximately -1.9 mV/°C.

The results of testing the Rev. 2 temperature sensor in water are shown in Figure 6.13. The sensor response was again linear, but also drastically improved in the sense that a smaller change in temperature resulted in a larger change in output voltage. The output changed with temperature by 9.6 mV/°C. Only a portion of the possible temperature sensing range is shown in Figure 6.13, but the response of the sensor will be approximately linear for temperatures in the entire range applicable to drinking water. The Rev. 1 test results show the diode sensing capabilities at a higher temperature range (up to 110°C) but it would also be beneficial to acquire a refrigeration system to fully characterize the device down to 0°C or below.

![Series-Diodes Temperature Sensor](image)

Figure 6.13. Rev. 2 temperature sensor results.
6.4 Level Sensor Test Results

The purpose of the water level sensor is to ensure that the chip is actually submerged in water when measurements are taken. Characterizing the water level sensor did not involve taking parametric sweeps. The signal conditioning circuitry was designed such that the output is either high or low depending on the value of the capacitor. The oscilloscope screen capture in Figure 6.14 illustrates the basis of the level sensor signal conditioning circuitry, which is that an RC integrator with a sufficiently large $\tau$ will result in a partially charged capacitor. The maximum output voltage can then be related to the value of the capacitor sensor.

![Oscilloscope screen shot showing that the RC integrator circuit does not fully charge to the 3.3 V input voltage.](image)

The capacitance of the sensor was measured with an LCR meter. The capacitance was 182 pF in air and 230 pF in water. Correct functionality of the capacitive level sensor and signal conditioning circuitry was verified.
Chapter 7

Conclusions

Two revisions of the multi-sensor chip were successfully designed, fabricated, and tested. The second revision of the chip delivered significant improvements over the first revision due to modifications in the sensor design and fabrication process flow. When implemented with signal conditioning circuitry, the turbidity, total dissolved solids, temperature, and water level sensors each produce a DC voltage output between 0 V and 1 V.

The intended application for this project is to be used as part of a system that indicates when a good source of drinking water has gone bad. The sensors are intended to be interfaced with a microprocessor that is capable of performing necessary signal analysis such as temperature compensation of the turbidity and TDS sensors. Each sensor chip would need to be calibrated to account for distributions in device characteristics due to process variations.

The performance of the sensor chip partially depends on the system surrounding it. For example, the placement and stability of the IR LED source has a great deal of impact on the turbidity sensor. Turbidity standard solutions should be used to calibrate or adjust the response of the turbidity sensor relative to the intensity of its IR source. Similarly, the performance of the chip relies on the stability and performance of the signal conditioning circuitry. Drifting power supplies could translate into fluctuating gain or DC offsets, which could then appear as a drift in one or more quality parameters.
The fabrication process used for the Rev. 2 wafers was new and uncharacterized. The motivation was to design a process that was customized for the multi-sensor chip, was time and cost effective by using minimal process steps, and was more predictable and robust than the RIT MEMS process. Shallow p-n junctions, designed for the benefit of the turbidity sensor photodiode, were made possible by the well controlled ion implant doping technique. Building the devices into n-wells rather than directly into the substrate allowed diodes to be connected in series for a stronger temperature sensor response. Switching to chromium metal proved to be worthwhile when the electromigration and corrosion issues that plagued the Rev. 1 TDS sensor did not appear to affect the Rev. 2 TDS sensor. The entire process required thirty-two steps, or half of the steps required for the RIT MEMS process.

There are many growth opportunities for this project. As well as working independently, the sensors should be verified to work together at the system level. Sensor calibration and temperature compensation techniques require further development, and interfacing a microprocessor for signal analysis will be a considerable task. Still, the results of this project show that MEMS technology can be used to create sensitive, compact, and inexpensive sensors for monitoring the quality of drinking water.
REFERENCES


## Appendix A

### Rev. 2 Fabrication Process Flow

<table>
<thead>
<tr>
<th>Date</th>
<th>#</th>
<th>Process Step</th>
<th>Recipes &amp; Conditions</th>
<th>Comments &amp; Measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP 10-Feb</td>
<td>1</td>
<td>P-type Wafers</td>
<td>D1-D6: 4-Point Probe</td>
<td>~576 um thick, 4 pt probe recipe &quot;6-inch bulk 1pt (semitool) Resistivity ~40 ohm-cm, D1 = 40.19 (center), 40.44 (edge), D2 = 41.04 (eddy current tool gave 23.2 ohm-cm)</td>
</tr>
<tr>
<td>HP 10-Feb</td>
<td>2</td>
<td>RCA clean</td>
<td></td>
<td>Normal</td>
</tr>
<tr>
<td>HP 10-Feb</td>
<td>3</td>
<td>Grow 1000A screen ox</td>
<td>Tube 1, recipe 311, 900°C.</td>
<td>Avg = 1033 A (dark violet) Range = 983 A to 1058 A</td>
</tr>
<tr>
<td>HP 15-Mar</td>
<td>4</td>
<td>Level 1 Photo: N-well</td>
<td>Coatml, 200 mJ/cm², 0.24 um, Devmtl</td>
<td>ST R093WATSEN_NWEL No alignment/resolution verniers</td>
</tr>
<tr>
<td>HP 15-Mar</td>
<td>5</td>
<td>Implant N-well</td>
<td>P31, 9E12, 120 keV</td>
<td>11 uA, 9E12 cm-2, 120 keV, 25 sec/wafer</td>
</tr>
<tr>
<td>HP 15-Mar</td>
<td>6</td>
<td>Etch 1000A Oxide</td>
<td>BOE. Test etch rate.</td>
<td>10:1 BOE, etch 2 min</td>
</tr>
<tr>
<td>HP 16-Mar</td>
<td>7</td>
<td>Ash resist</td>
<td>Branson asher</td>
<td>6&quot; normal ash. 1 wafer had to go back in</td>
</tr>
<tr>
<td>HP 16-Mar</td>
<td>8</td>
<td>RCA clean</td>
<td></td>
<td>Normal</td>
</tr>
<tr>
<td>HP 17-Mar</td>
<td>9</td>
<td>Well drive-500A oxide</td>
<td>24 hrs 1100C N2, 17 min 1100C dry O2 (recipe 19)</td>
<td>Tube 1 - ~1000A in nwell (dark violet-blue), 1500A field ox (light metallic blue)</td>
</tr>
<tr>
<td>HP 23-Mar</td>
<td>10</td>
<td>Level 1,5 Photo: Channel Stop</td>
<td>120 mJ/cm², 0.24 um, Devmtl (Nwell mask)</td>
<td>ST R093WATSEN_NWEL Reverse pattern/Negative resist - AZ nLOF 2020 - spin 3000 RPM for 30 sec (SCS spin coater), bake 95C for 90sec, thickness on spectramap FT500 (OIR recipe) ~12,662 A</td>
</tr>
<tr>
<td>HP 23-Mar</td>
<td>11</td>
<td>Implant Channel Stop</td>
<td>Boron, 8E13, 80 keV</td>
<td>I<del>55 uA, t</del>45 sec/wafer, B11, 8E13, 80 keV</td>
</tr>
<tr>
<td>HP 23-Mar</td>
<td>12</td>
<td>Ash resist</td>
<td>Branson Asher</td>
<td>6&quot; hard ash</td>
</tr>
<tr>
<td>HP 17-Mar</td>
<td>13</td>
<td>Level 2 Photo: P+</td>
<td>Coatml, 200 mJ/cm², 0.24 um, Devmtl</td>
<td>ST R093WATSEN2_2 Perfect alignment, 1 um resolution</td>
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<tr>
<td>HP 17-Mar</td>
<td>14</td>
<td>Implant P+</td>
<td>Boron, 1.8E13, 45 keV</td>
<td>1.8E13, 45 keV, B11, ~30 sec/wafer (20 uA)</td>
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<tr>
<td>Date</td>
<td>Step</td>
<td>Description</td>
<td>Details</td>
<td></td>
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<tr>
<td>18-Mar</td>
<td>Ash resist</td>
<td>Branson Asher 6&quot; hard ash</td>
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<tr>
<td>18-Mar</td>
<td>Level 3 Photo: N+</td>
<td>Coatmtl, 200 mJ/cm², 0.24 um, Devmtl ST R093WATSEN2_2 Perfect alignment, 1 um resolution</td>
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<tr>
<td>19-Mar</td>
<td>Implant N+</td>
<td>Phosphorus, 3E15, 60 keV 3E15, 60 keV, 160 uA, 600 sec/wafer - photoresist is dark</td>
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<tr>
<td>18-Mar</td>
<td>Ash resist</td>
<td>Branson Asher 6&quot; hard ash - twice</td>
<td></td>
<td></td>
</tr>
<tr>
<td>24-Mar</td>
<td>RCA clean</td>
<td>Normal</td>
<td></td>
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<tr>
<td>26-Mar</td>
<td>Anneal/Grow 3500A Oxide</td>
<td>1000C, 44 min, wet O2 (recipe 400) Tube 1 - Avg = 3500 A</td>
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</tr>
<tr>
<td>26-Mar</td>
<td>Level 4 Photo: CC</td>
<td>Coat, 200 mJ/cm², 0.24 um, DevCC ST R093WATSEN2_2 Perfect alignment, 1 um resolution</td>
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<tr>
<td>26-Mar</td>
<td>Contact Cut Etch</td>
<td>wet etch 3500 A 385 A/min - 1tch 10.5 min</td>
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<tr>
<td>26-Mar</td>
<td>Ash resist</td>
<td>Branson Asher</td>
<td></td>
<td></td>
</tr>
<tr>
<td>26-Mar</td>
<td>RCA clean</td>
<td>Extra HF dip (30 sec) D1-D3 + dummy</td>
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<tr>
<td>5-Apr</td>
<td>Deposit Metal</td>
<td>Sputter 5000A Chromium pre-sputtered to get rid of arcing. dep rate ~350 A/min, 5 mTorr Argon, 1350 Watts, flow = 20, time = 15 min, AlphaStep ~4kA to 5kA</td>
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</tr>
<tr>
<td>12-Apr</td>
<td>Level 5 Photo: Metal</td>
<td>Coatmtl, 180 mJ/cm², 0.24 um, Devmtl ST R093WATSEN2_2 Perfect alignment, 1 um resolution</td>
<td></td>
<td></td>
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<tr>
<td>12-Apr</td>
<td>Metal Etch</td>
<td>Etch 5000A Chromium Chrome etch ~10 min, 1/2 micron undercut</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14-Apr</td>
<td>Strip Resist</td>
<td>Solvent Strip Use PRS-2000 solvent strip!!!!</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15-Apr</td>
<td>Deposit TEOS</td>
<td>Deposit ~7000A LTO. D1-D2, LTO - 156A/min, ~6000 A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15-Apr</td>
<td>Level 6 Photo: Via</td>
<td>Coat, 180 mJ/cm², 0.24 um, Develop ST R093WATSEN2_2 D1-D2</td>
<td></td>
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<tr>
<td>16-Apr</td>
<td>Etch Oxide for Vias</td>
<td>Wet Etch 6000A</td>
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<td>16-Apr</td>
<td>Strip Resist</td>
<td>Solvent Strip</td>
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<tr>
<td>33</td>
<td>Test!!</td>
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