Internship Report
Mandatory Internship at Texas Instruments

Development of a wireless operated chemical sensor prototype

KILBY Lab Freising

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I certify that this report is my own work, based on my personal study and research during my internship at Texas Instruments.

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Abstract

The pH value, i. e. the ion concentration in a solution, is an important factor in our daily life. You're dealing with pH values when it comes to water quality, pool chemistry, wound healing, laundry and much more. A very attractive way to measure pH value is to use a so-called ISFET (ion-sensitive field-effect transistor).

Kilby Lab Freising has been developing such e-sensors in cooperation with Freising fab for some time and now and the first chips are out for testing. To demonstrate the potential of this new silicon, a possible application was implemented and evaluated: A NFC-powered chemical sensor tag.

This paper is about the implementation of an ISFET sensor and development of the final tag. I will focus on the challenges I faced during development, such as optimizing power consumption, packaging the sensor, and dealing with lifetime problems of this rather early sensor. The final tag is fully operational and can be read out by an Android App.

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1 Introduction

Today, a variety of pH sensible sensors are available on the market. Most of them use a combination of a reference electrode, combined with a second metal electrode that changes potential in correlation to a change in ion concentration. Another method is to use a ISFET as the ion-sensing element. These components also require a reference electrode to achieve absolute pH measurement.

In this paper we demonstrate a possible application for a new ISFET that is currently developed by Texas Instruments. The goal is to produce a sealed sensor with a volume of $1cm^3$ that measures the absolute pH in solution, powered and read out by a common smartphone.

The sensor used for this demo was fabricated 2017 by Texas Instruments in Freising. It contains 4 different ISFET (ion-sensitive field-effect-transistors) designs, 4 complementary REF-FETs (reference field-effect-transistors) and two platinum electrode structures. At least two different ISFET should be read out via the smartphone, using on of the REFFETs for compensation.

2 Design

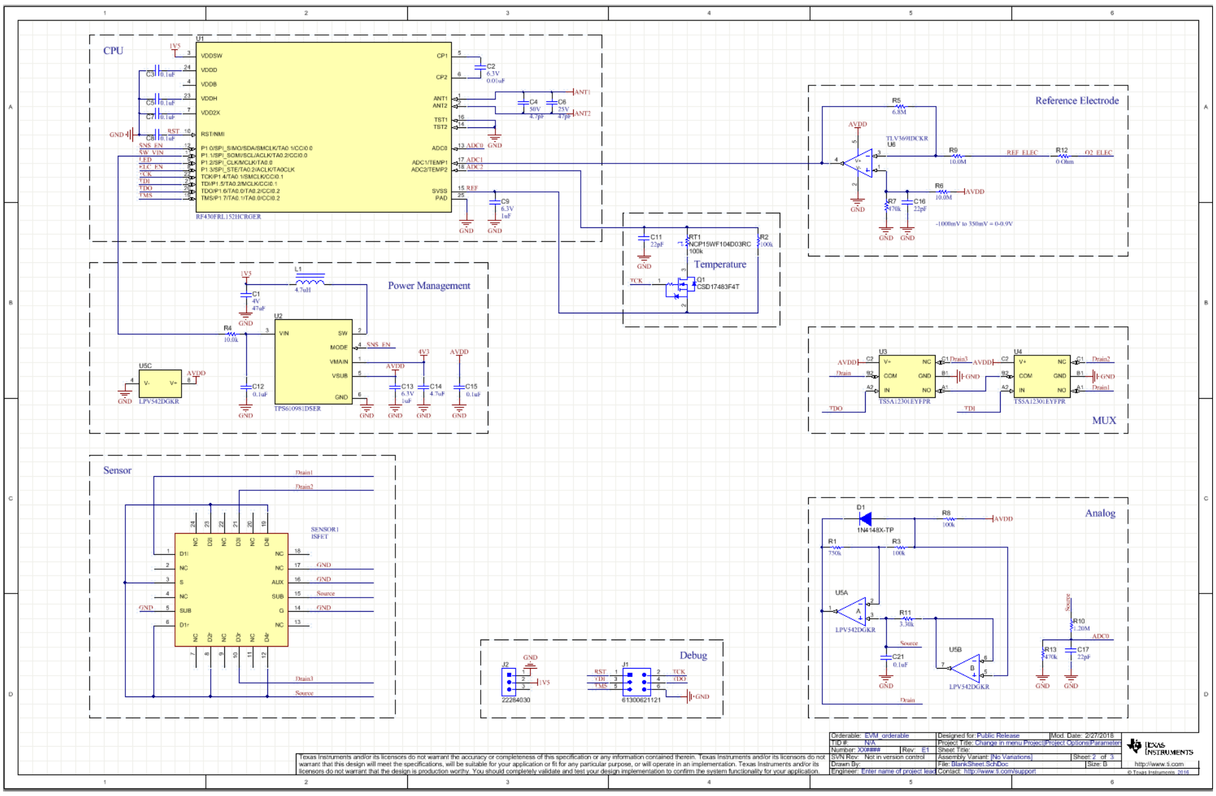


Figure 1: Schematics of the final sensor tag.

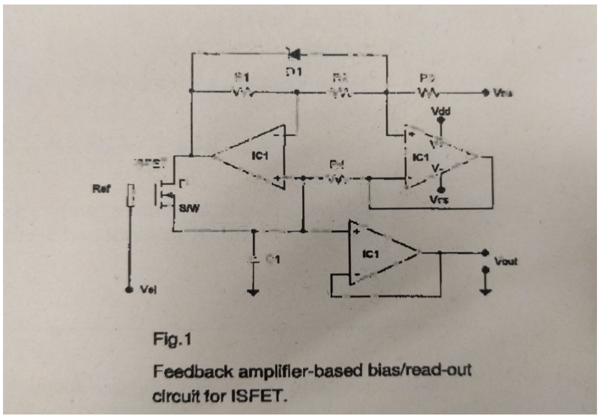
2.1 First analog design considerations

An ISFET basically acts like a common MOSFET, however, the Gate-Threshold Voltage varies over ion concentration. It is build in a way that allows the ion-sensitive gate isolator to get in touch with the liquid of interest. An external electrode is used as the actual gate connection, so that the liquid has a known potential.

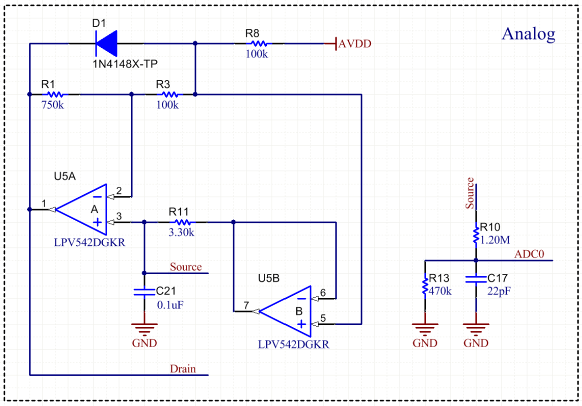
Same as for MOSFET, p-channel and n-channel type ISFET exist. As for my research only p-type ISFETs were available, I used these for designing a readout circuit.

In order to only measure the change of ion concentration, all other parameters that have an effect on the threshold Voltage must be controlled. For this, a circuit is required that applies constant drain-source current on the ISFET. Basically this can be achieved by a simple constant current source that drives the gate electrode for feedback. However as this is a p-type FET, a negative power rail would be required.

A more practical circuit found in literature uses two OpAmps to create a “floating” current source. The gate electrode of the ISFET is at a fix potential of 0V, the constant DS current flow is set by variating the Drain and Source Voltage against ground.



Schematic from literature



Power-optimized circuitry

To allow the sensor to be powered by NFC, the current consumption must be as low as possible. For this reason, the low Iq (quiescent current) OpAmp LPV534 was used as the OpAmp. Also the 2.5V voltage reference diode was replaced by a 4148-type Si-diode with a voltage drop of 0.7V. This allows operation voltage to go down to as low as 2V.

As this circuit is meant for measuring the Gate-Threshold Voltage, the OpAmp output swing needs to cover the Gate-Threshold Voltage of the ISFET under all operation conditions. The Gate-Threshold Voltage of the used ISFETs was found to be in the range of 1.2V – 2.7V. As the LPV534 is a Rail-to-Rail Operational Amplifier, a positive power supply of 3V is enough to sufficiently power this circuit.

2.2 First digital design considerations

One of the goals of this project was to not only power this sensor by NFC, but also to read the sensor data using smart phone. Therefore some sort of microcontroller is necessary that handles sensor readout.

The RF430FRL152h is a MSP430-based IC combined with a 13.35MHz ISO15693 transponder AFE. This chip offers a lot of features that come in handy considering the total sensor design:

- Can be powered by a battery or entirely by a NFC reader
- Supports the ISO15693 protocol to transmit data to the reader.
- 3 ADC channels with up to 14bit resolution

- Internal rectification of the NFC field, 1.5V regulated core voltage + 2.9V doubled field voltage externally available.
- Fully programmable
- Enough GPIOs for additional circuity such as MUX control inputs.

The MSP430 core can be programmed via JTAG. However, most of the microcontroller's FRAM is read- and writable via NFC which allows data exchange via memory-mapped registers, but also the possibility of over-the-Air firmware updates. For most of the time, the JTAG interface was used for programming and debugging.

The RF430FRL152h has some ROM functions that are programmed during manufacturing. This ROM functions can be called via NFC and allows to for example take ADC readings or toggle GPIOs without having to actually program the IC. However these functions are very limited and for flexibility reasons there were no attempt to fully base the application firmware on these ROM instructions.

2.3 First power design considerations

The first idea was to use the 2.9V provided by the RF430 to power the ISFET readout circuity, as suggested by application examples in the datasheet. However unfortunately this internal voltage doubler can only supply 100µA. Also this voltage is not stabilized and therefore not suitable to power the ISFET.

An external circuit was required to push the available 1.5V to a 3V voltage level. The TPS610981 is a very small low power boost converter IC with integrated 3V LDO. This offers not only a high, but also very "clean" and stable power supply to power the OpAmps used in the sensor.

An Keythley 3624A Sourcemeter was used to get a first idea on how much power was available from the NFC field of a common smartphone. For this, a RF430 evaluation board was programmed to send out a heartbeat to a smartphone. The Nexus 5X smartphone was then placed at various distances over the NFC antenna of the EVM. Then the 1.5V rail was drained by increasing current until the microcontroller passed out and sent out no further heartbeats.

Distance	UVLO: 1.5V	UVLO: 1.4V
30mm	0µA	140µA
25mm	100µA	600µA
15mm	170µA	760µA
5mm	70µA	1130µA

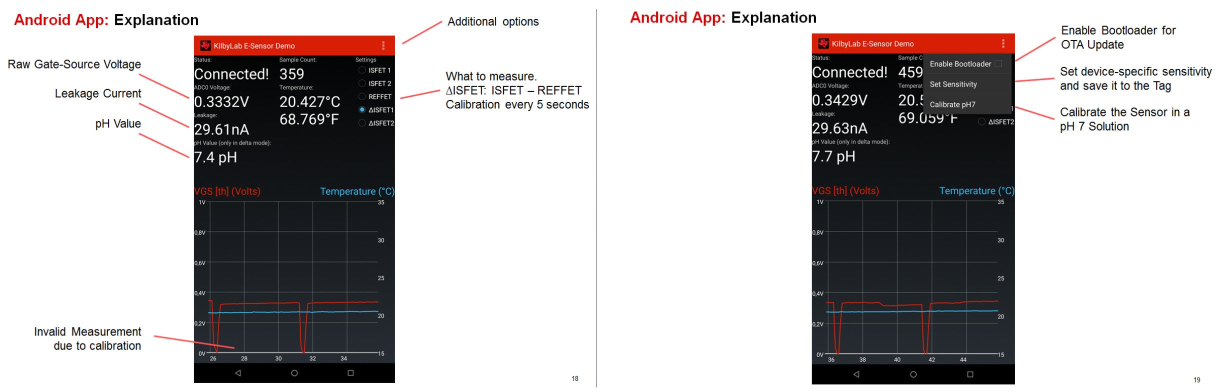
At an undervoltage lockout of 1.4V (barely the minimum required for proper operation), a current of 700µA can be drain while sustaining a sufficient distance.

2.4 Android App

An Android App is used as the main software to read out the sensor. The problem with NFC in Android is that no direct control of the NFC transceiver chip is possible. Instead, Android abstracts all the specific chip function into a generic NFC interface, that can be commanded to schedule read and write request via NFC. Unfortunately, this forbids to leave the NFC field switched on without communication happening, which is required to fulfill the high power requirement of the sensor tag. Also, no signal quality information can be obtained that would be useful to estimate maximum transmit distance.

To overcome this problem, read request must be perfromed with maximum possible rate, even if no new samples are available. This ensures the NFC field stays on and continuously powers the sensor.

For example, the sensor performs ADC sampling and updating of the memory-mapped registers with about 5Hz. However, the used Nexus 5x running Android 8.1.0 is capable of 20-25 NFC read requests per second. Another advantage of this oversampling is that even as the data is polled asynchronously, no data loss can occur.



The whole communication protocol between tag and reader uses three ISO15693 blocks with 8 bytes block length.

- 1. Block: Firmware version + calibration data (offset + sensitivity)
- 2. Block: ADC data, 2 bytes per 14bit reading
- 3. Block: Status bytes, bootloader flag

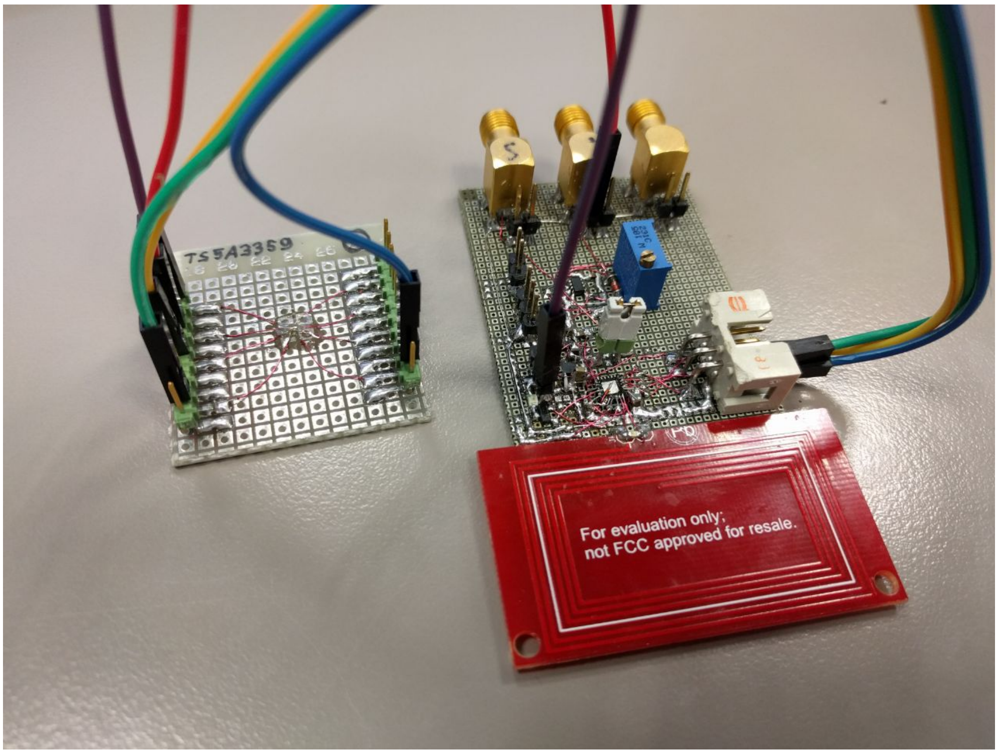
The Status byte is used for bidirectional communication. Writing specific bits to this register changes eg. the selected ISFET by switching the MUX.

As the RF430FRL152h uses FRAM, all code and register values are persistent even without power.
Setting the bootloader bit stops the usual power up sequence of the tag and ensures the tag is ready to write a new firmware over the air. No actual bootloader is required for this as all relevant code is in the NFC-writeable flash area, however flashing the tag in normal operation can cause interferences that make successful flashing impossible. The bit is cleared after a successful OTA update, or by itself after 3 minutes in case the bootloader was activated by accident.

3 Development

3.1 First Prototype: Lessons learned

After building the first prototype and ensuring the actual RF430 works and executes code, trying to power up the TPS610981 boost converter was unsuccessful at first try. As turned out, this chip requires a very low impedance of the power source as it draws about 20mA for about 50us during startup sequence. After that, current consumptions goes down to the expected 200nA. To fulfill this current requirement, large tank capacitor of 47uF what added at the input of the boost converter circuity. When putting the tag into a NFC field, the startup of the DCDC must be verzögert by few seconds to allow the tank capacitor to charge up to 1.5V. As the TPS610981 has no dedicated enable pin, the Vin pin is connected to a GPIO of the RF430. Once the TPS610981 started up successful it powers itself and the GPIO can be configured as an input to safe few Nanoamps.



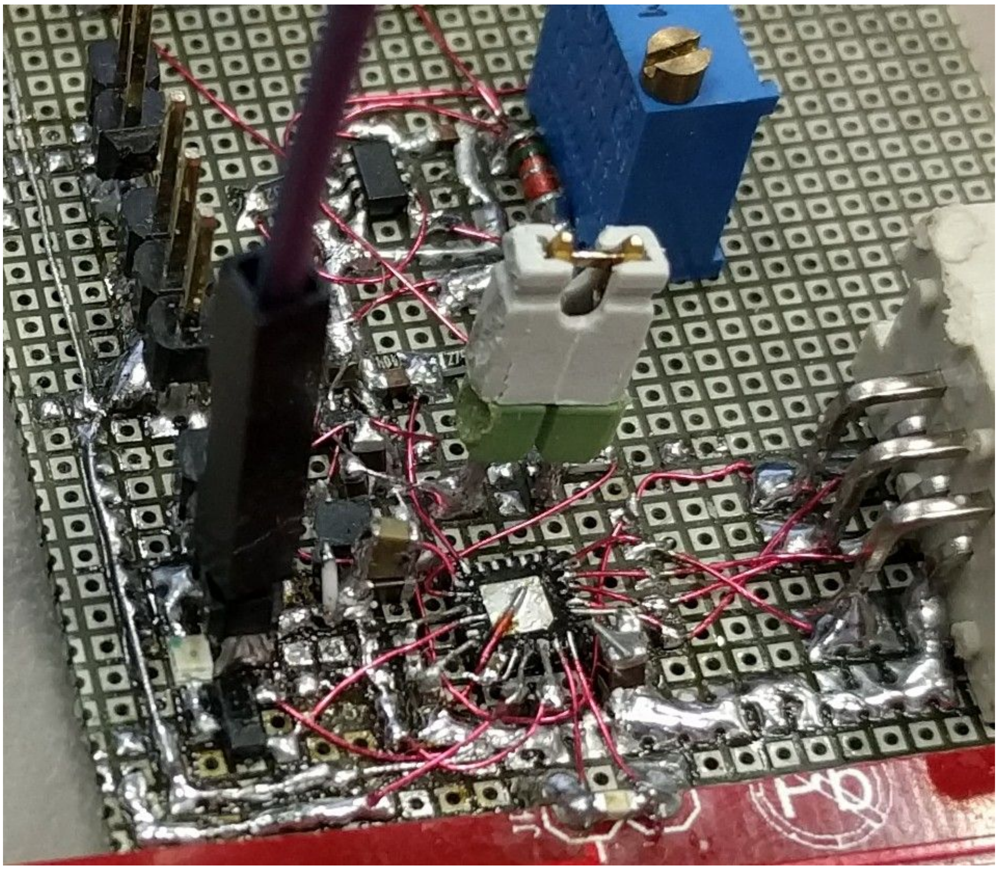


Figure 2: First prototype

3.2 Final sensor packaging considerations

TI already produces NFC tags, called laundry-tag as these are used to track laundry. The original laundry-tag is a passiv NFC tag that is molded into a 20mm plastic disk. This makes it waterproof and very robust to a variety of chemicals. For manufacturing of the laundry tag, the NFC parts are placed in the bottom half of the casing, which is then injection-filled to complete the package.

The same packaging should be used for this smart sensor. As this would cover the sensor, only the lower half of the laundry-tag package is used while the upper half needs to be applied by hand.

After testing multiple possibilities of the exact procedure, the best way to package the sensor turned out to be the following:

- 1. A thin, but still solid pcb including the NFC antenna is assembled from one side.
- 2. In the middle of the backside, the sensor die gets bonded right onto the pcb (chip on board).

- 3. Using epoxy, a dam is build around the chip by hand. It covers the bond wires, but not the whole sensor nor the complete pcb.
- 4. The Sensor module is placed with components on the downside in a milled out bottom half.
- 5. The void between the epoxy dam and the edge of the bottom half is filled with epoxy till everything except for the active sensor area is covered.

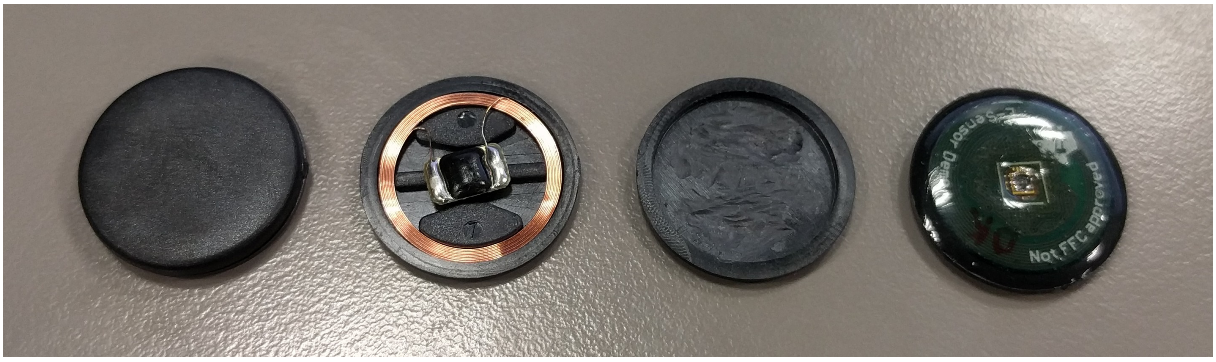


Figure 3: Left to right: laundry tag; opened laundry tag; milled-out bottom half; finished sensor tag

Due to the bad quality of the dies used for this project, we later decided that it would be best to first bond the chip onto the pcb and then do the assembly. This allows to probe the bonded chip using a 3D printed bed of nails, and saves populated, but not working pcbs.

3.3 Second Prototype layout considerations

The maximum space available inside the laundry tag case is a disk with 18mm diameter and 2mm height. This is rather small for the amount of components that have to fit in there. In addition some area is wasted as it is not recommended to place components above the pcb antenna coil . This leaves about 12mm for the microcontroller, analog circuitry and power. To make this possible, 0201 passive component size was chosen. This is still big enough to be assembled by hand, but safes space compared to the much easier to handle 0402 packages.

Even through the RF430 is programmable over-the-air, a JTAG breakout header was placed on one side of the pcb. This header can be removed after testing and safes a lot of trouble while debugging.

As the whole circuity is directly exposed to the strong, high frequency NFC field, analog design is critical. A 22pF capacitor was placed as close as possible to the ADC inputs to minimize interference.

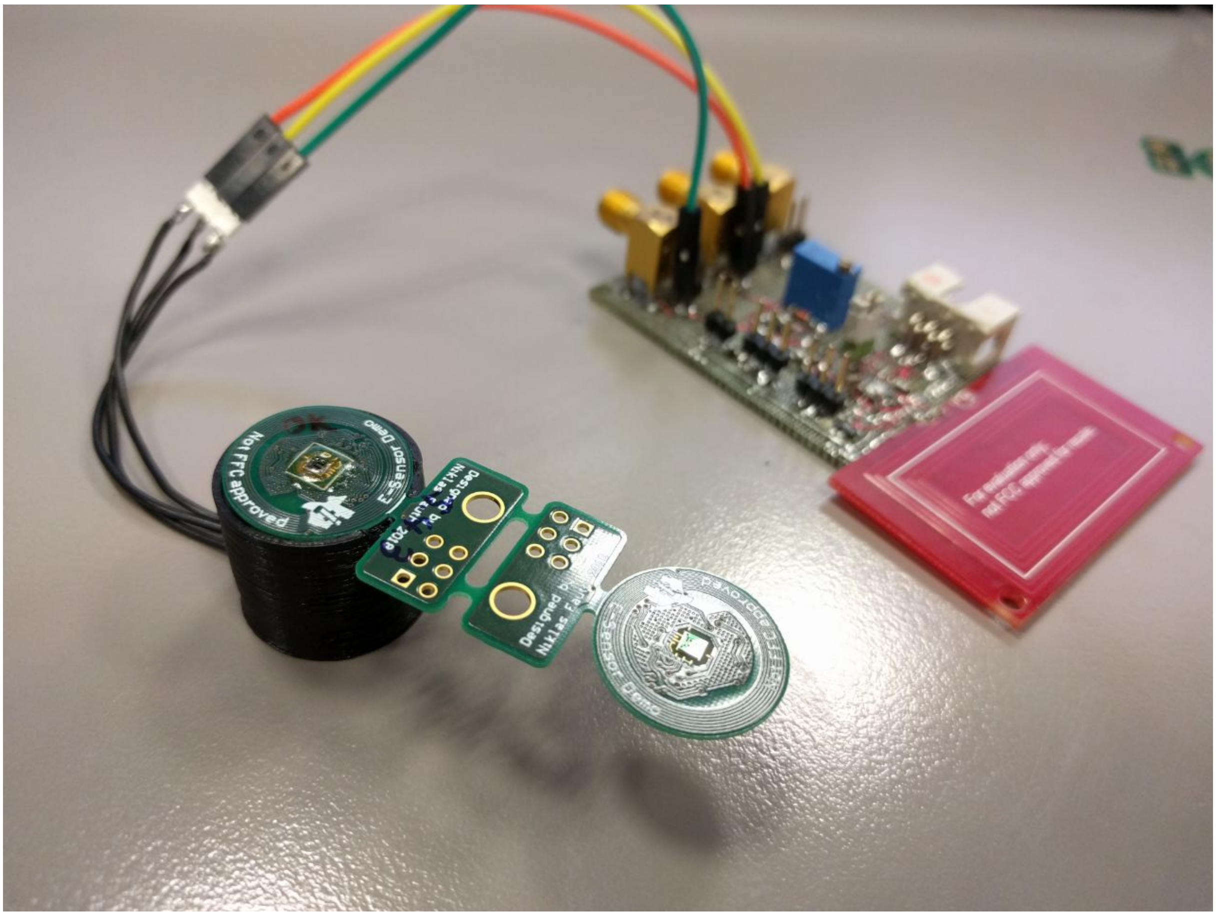


Figure 4: Testing the bonded ISFET dies using a bed of nails

The ground plane on the senor side was designed as hatched pour, as this reduced capacity between layers and has increases planar inductivity, which reduces eddy currents that weaken the NFC field.

3.4 Second Prototype assembly

For testing, a total of 6 pcbs were assembled by hand. First all SMD components were placed and reflowed on the component side of the sensor. To tune the NFC antenna for maximum distance the assembled sensor pcb was placed inside a ring probe, attached to a network analyzer in S11 mode[1]. There are two 0201 footprints for tuning capacitors, so that the capacitance can be fine tuned by using a bigger capacitor (47pF) in parallel with a smaller one (2pF) for fine tuning.

Now the sensor die got bonded onto the sensor pcb by wire bonding. For this, a special mount has been crafted that fits the assembled pcb for the wire bonder mount. The sensor was bonded using the "stich on bump" method.

[1] http://www.st.com/content/ccc/resource/technical/document/application$_{n}ote/d9/29/ad/cc/04/7c/4c/1e/CD00221490.pdf$

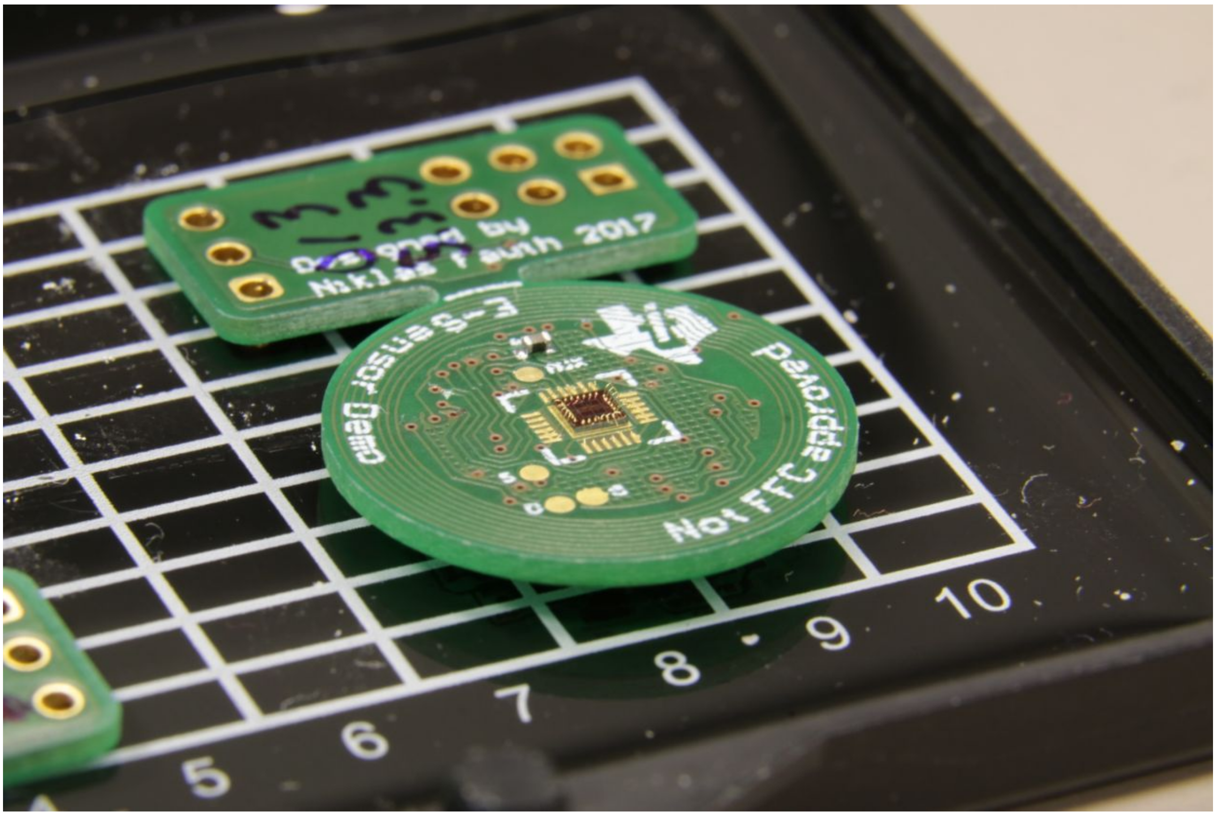


Figure 5: Bonded die on pcb

After successful bonding, the bonding wires were sealed with epoxy. It took some training with broken dies to get good results on the final pcbs. “Uhu Endfest 2k” Epoxy was used for this, as it hardens within just 2min. This gives fine control on the viscosity of the glue by adjusting timing. Basically, the trick is to use a fine wire to apply a droplet of glue around the sensor onto the pcb.

The glue spreads towards the edges of the sensor die, and once the glue has hardened enough is was gently pushed towards the sensor die again with said wire. This forces the epoxy over the top edge of the die, covering the bonding wires down to the pcb. If you didn’t wait enough, the epoxy is too viscous and spreads over the whole die surface, rendering the sensor unusable. If you wait too long for this step, the epoxy already hardened and it doesn’t cover all conducting surfaces like bonding pads, or even breaks bonding wires when trying to push it too hard.

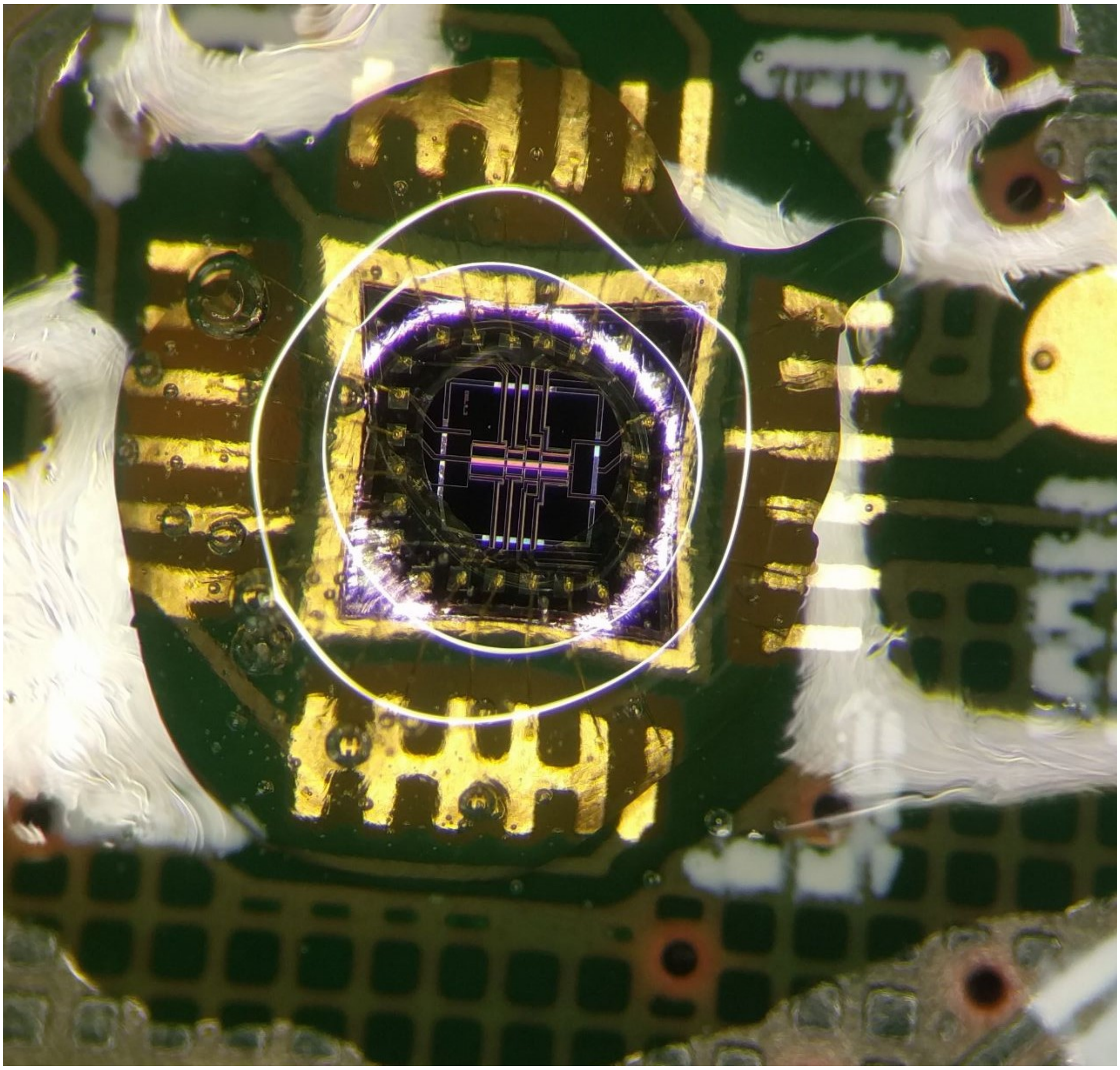


Figure 6: Successfully sealed bond wires

3.5 Second Prototype: Lessons learned

As most of the circuitry was tested using the first prototype, it was no surprise to find most working as expected. The sensor delivered some plausible readings. However when trying to switch the MUX in order to read out a different ISFET, the NFC connection fails and the sensor gets lost.

The MUX Ic was the only thing not tested before, as it was expected to work out of the box. However closer investigations on this IC on a separate breakout board revealed that this particular IC causes current spikes of up to 2mA whenever the switch setting is changed. This is not documented in the data sheet as it is not a problematic behavior for general purpose applications.

However this current spike causes similar problems to those of the DCDC converter while powerup: As NFC works by field strength modulation, a rapid change in current consumption also modulates the NFC field strength in a unwanted way and can cause transmission errors that can lead to a loss of the connection.

To fix this problem, a analog switch with constant threshold voltage was used instead of the TS5A3359. [2]

Unfortunately, only the TS5A12301E , which is a 2:1 switch, supports this feature. To get three channels for reading three different ISFETs, two TS5A12301E have to be put in series. To test if this works, two TS5A12301E were soldered on the second prototypes. Test were successful, so this configuration is used for the third prototype.

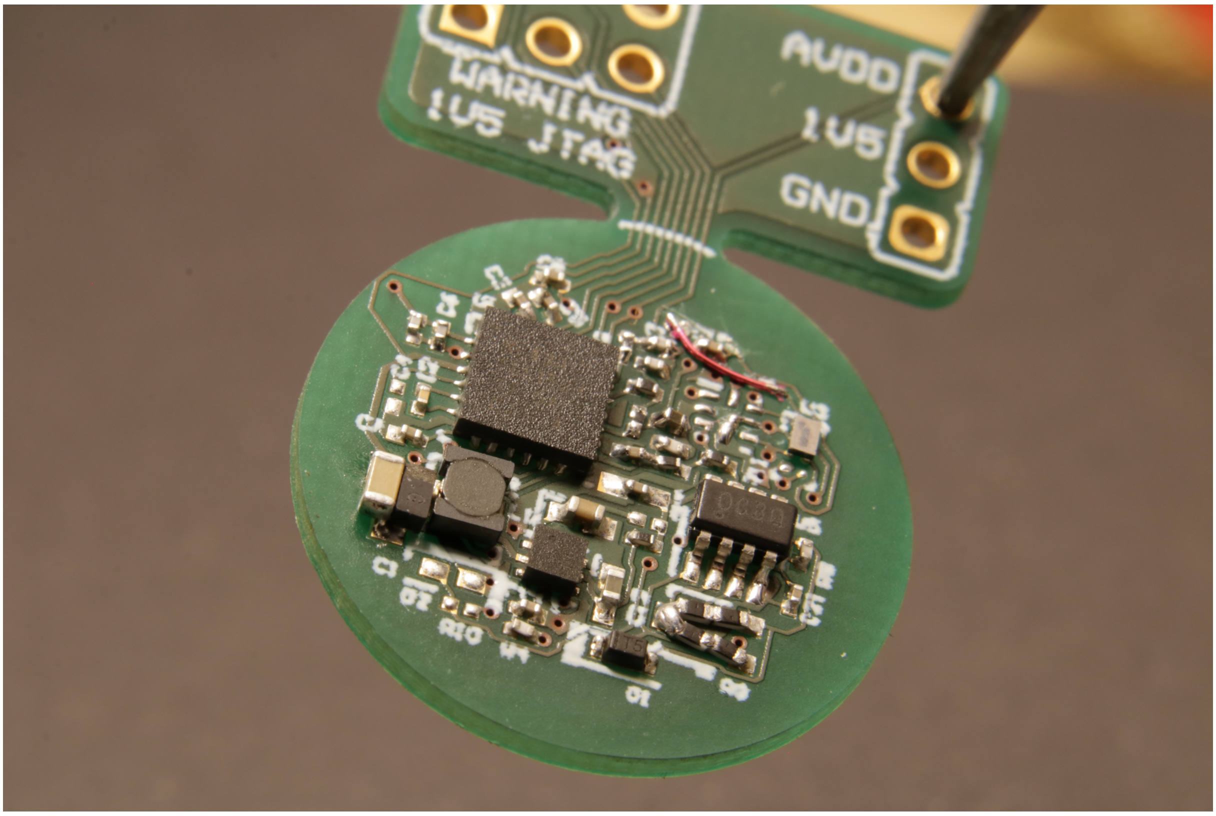


Figure 7: Second prototype with bodged analog MUX

[2] http://www.ti.com/lit/an/scda011/scda011.pdf

3.6 Leakage detection

As it turned out, the wafer used for this projects has some technical teething troubles the need to be kept in mind while working with the sensor. One of them is the passivation layer, that shows leakage after a certain amount of time:

When applying 5V between source and the electrolyte, after 20 - 180min current starts leaking through defects in the passivation layer. This effect also occurs at voltages down to 2.1V, so using the sensor under normal conditions causes damage that limits lifetime.

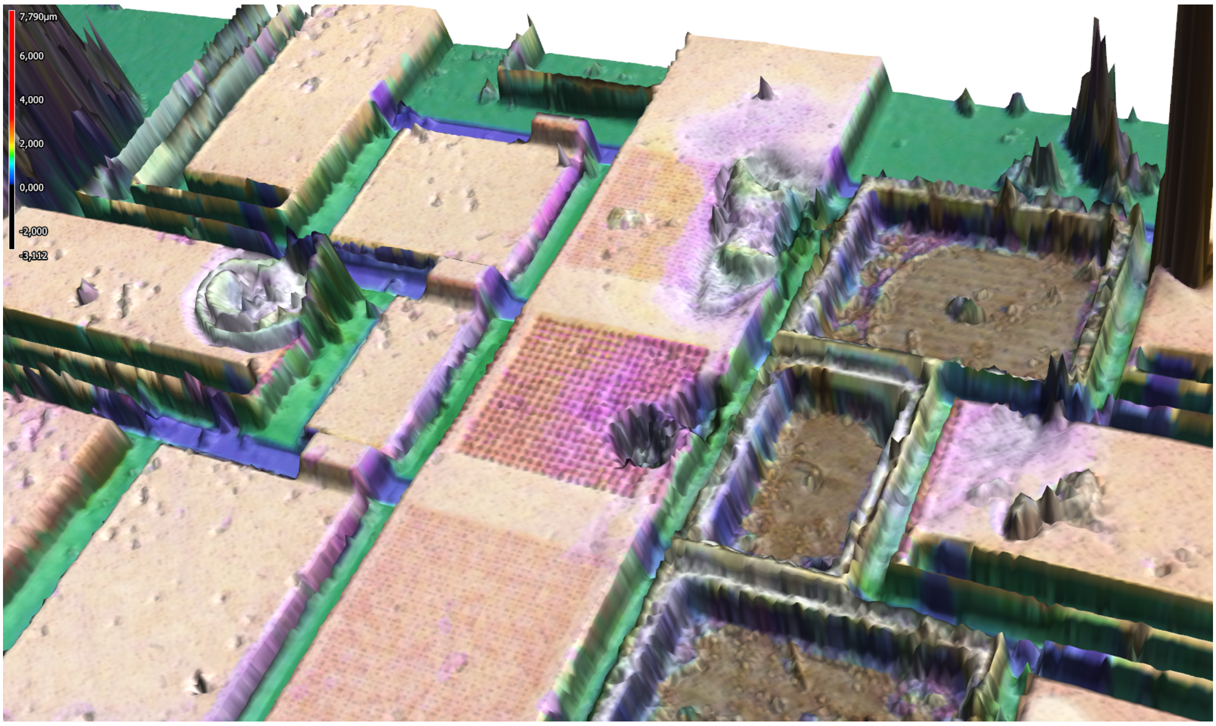


Figure 8: Holes in passivation after 3h of normal operating. 3D image created by laser depth measurement. conditions

At this point there is no solution for this problem. The next sensor generation will hopefully overcome this problem by using a thicker passivation layer, and lowering the gate-source threshold voltage, so lower and thus less harmful voltages can be used for operation.

Once current leaks in the electrolyte, the potential changes and the measurement is unusable. It is therefore necessary to detect leakage to ensure accurate measurements.

The ISFET sensor die contains two electrodes: One big platinum electrode that is far apart from the core containing the ISFET. This electrode is used for reference 0V potential. A second platinum electrode closer around the ISFETs was originally planned to apply voltage to the electrolyte for calibration and oxygen measurement. For this sensor tag application, this electrode is left floating. Measuring the voltage at this electrode can tell whether a leak occurred: If

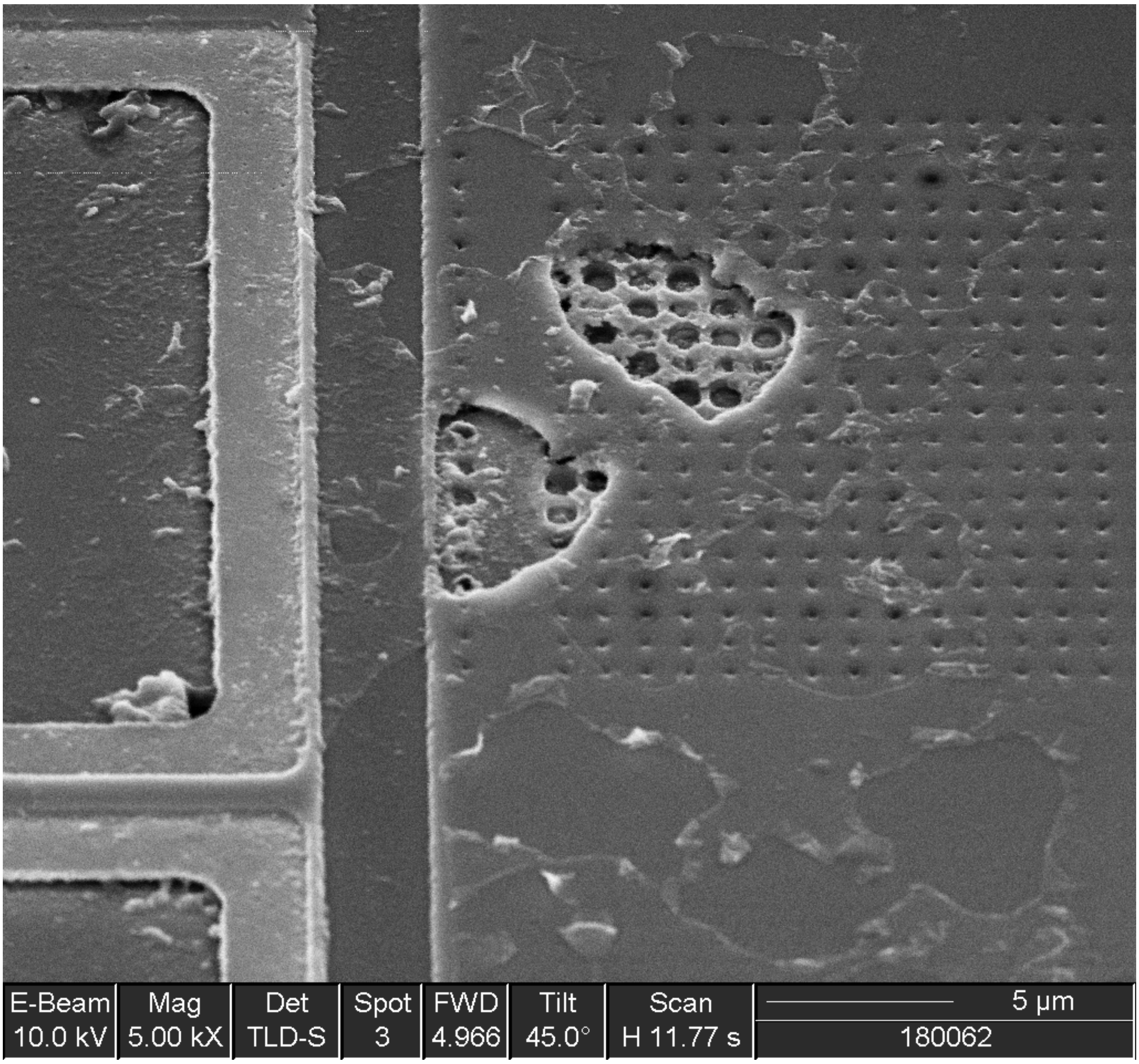


Figure 9: Detailed REM view of a defect caused by leakage

the potential is not 0V, a current leaks into the electrolyte and disturbs the measurement. This is detected by the sensor tag, the Android App shows a warning that the measurement might not be accurate.

4 Result

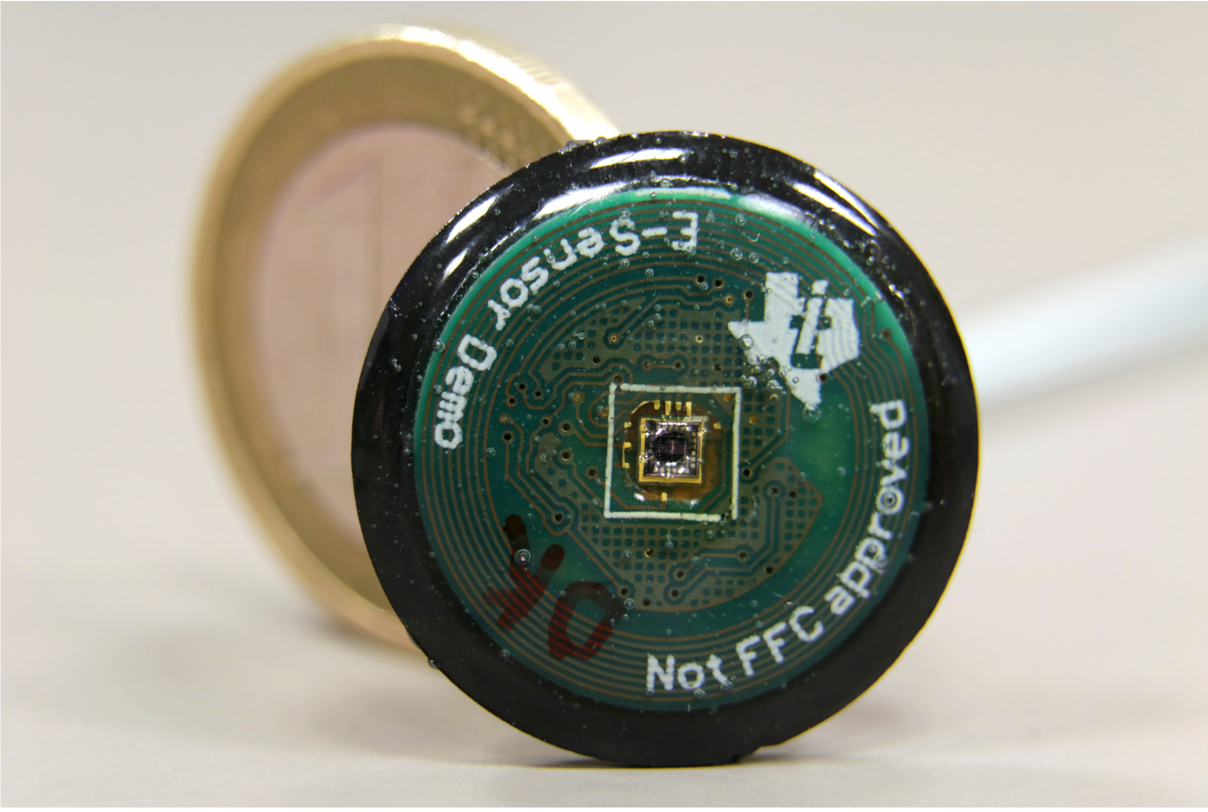


Figure 10: Final sensor tag

In total, eleven final sensor tags have been build (by hand). All of them work fine, pH measurement using the App is possible. Even if the lifetime is limited to few hours, these sensor tags impressively demonstrate the potential of these new sensor ICs. The pinout of the sensor die won't change for the next revision, so the design can be reused for the next generation of sensors that might not suffer from the lifetime problem and work perfectly fine for years.

Figure 11: Sensortag in solution, connected to a smartphone using a self build NFC expander

Figure 12: Final sensor tag