A high sensitivity open loop electronics for gravimetric acoustic wave-based sensors

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Abstract: in order to use radiofrequency acoustic transducers as gravimetric sensors for detecting chemical species in gas phase, we propose an openloop interrogation strategy. As opposed to the closed loop – oscillator – strategy whose performance is strongly dependent on the environment of the acoustic transducer and might yield to a lack of oscillation if the damping of the acousic wave is not compensated for by the amplifier, an openloop strategy provides, beyond the actual acoustic parameters, the data needed for the diagnostics of a poorly performing transducer. Indeed, in an openloop strategy, the large dynamics of the I/Q demodulator needed to extract the phase and magnitude information at each frequency yields improved robustness of the measurement system – transducer and associated electronics – with respect to the closed loop strategy. However, reaching comparable noise levels with the former strategy is challenging: we propose an embedded frequency-sweep network analyzer dedicated to gas-phase monitoring of chemical compounds with improved sensitivy with respect to commercially available integrated circuits or general purpose network analyzers.

We demonstrate a 16 Hz standard deviation at 125 MHz, with a working frequency band in the 60 to 133 MHz range, answering the requirements of using Rayleigh and Love-mode acoustic 40 μm wavelength transducers as classically found in the litterature. The remaining noise source is solely due to the Analog-to-Digital Converter of the microcontroller, so room is left for improving this result by optimizing the voltage reference and board layout.

1 Introduction

Current systems for detecting potentially toxic gases are in genral cumbersome and complex. Aimed at continuous, real time monitoring, we shall focus solely on direct detection sensors which do not require preliminary sample processing: optical [1], electrochemical [2] or gravimetric acoustic sensors [3] have been demonstrated for such purposes. Most references in the litterature focus on improving the transduction principle [4, 5, 6], either by replacing propagating waves with evanescent waves which, by being confined close to the transducer surface, improve the signal to noise ratio (Surface Plasmon Resonance – SPR – and Love mode surface acoustic wave sensors – SAW), while we are interested here in developing the embedded electronics associated with the dedicated application of chemical species using radiofrequency (RF) transducers. One of the aspects of this dedicated application is the ability to identify an optimal frequency within band-pass range of the transducer, and stop the sweep in order to generate a clean, continuous wave signal at this given frequency, with sub-hertz resolution.

The basic principle of gravimetric acoustic wave sensors is the measurement of the phase velocity variation due to the adsorbed mass density or thickness change during a chemical reaction: this phase velocity is dependent on the boundary conditions of the propagating surface acoustic wave, and is affected either by the guiding layer density and/or thickness.

We here focus on the development of a dedicated, embedded instrument based on a full software control for the openloop monitoring of delay lines providing the same stability and accuracy than a closed loop approach. In order to reach this aim, a Direct Digital Synthesizer based oscillator provides the flexible RF probe signal, while a low noise phase detector is fed by an amplitude controled signal. This low noise electronics is used in parallel with a flexible wideband I/Q demodulator for the preliminary characterization of the transfer function of each new sensor and selecting the optimum working frequency used throughout the gravimetric detection experiment.

2 Basic principles

2.1 Radiofrequency signal controls

In order to probe gravimetric accoustic wave-based sensors we have to provide RF signals: we focus on working in the 60-133 MHz frequency range, consistent with the use of 40-μm wavelength Rayleigh wave or Love mode surface acoustic wave sensors. The RF probe signal is generated by an Analog Devices AD9954 Direct Digital Synthesizer, controled by an ARM7-core based ADuC7026 microcontroler through the SPI link. In order to perform differential measurements, two transducers are probed simultaneously, one coated with a sensing layer and the other one kept clean as a reference. Since the two devices exhibit different working conditions, either due to manufacturing differences or due to the sensing layer coating, we generate two independent probe signals using two DDS. Each RF signal is split between a reference channel – a programmable attenuator tuned to generate an output signal with a power close to that going through the SAW delay line used as measurement line.

A coarse measurement mode uses an integrated I/Q demodulator (Fig.1) – Analog Device's AD8302 – whose high bandwidth provides a noise level of $\pm 0.3^o$ on the phase detection output, consistent with the performance of most frequency-sweep RF network analyzers, providing an output similar to the S_{21} parameter classically used to characterize SAW delay lines.

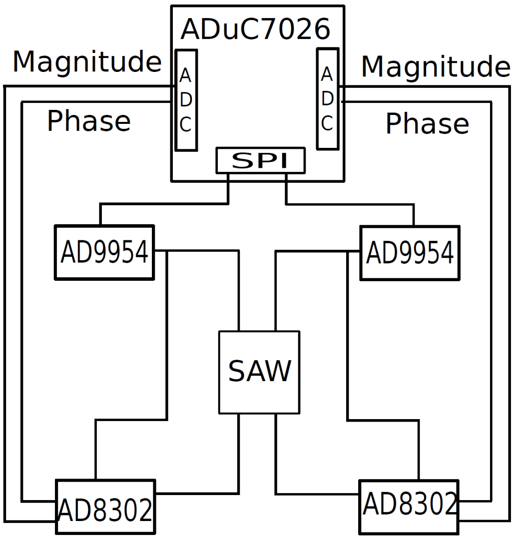


Figure 1: General diagram of the SAW-monitoring electronics, including an ADuC7026 microncontroller, AD9954 Direct Digital Synthesizer, and AD8302 I/Q demodulator.

The setpoint – selection of the frequency at which all measurements will be performed during the chemical reaction – is computed following a full-band frequency sweep and the identification of the maximum transmitted signal, max($|S_{21}|$). Having identified this setpoint, a search on the S_{21} phase identifies the closest mid-scale crossing condition, at which the I/Q demodulator exhibits the strongest linearity and at which the analog to digital converter provides the widest dynamics. Having finely tuned the frequency, we fix this setpoint throughout the chemical reaction monitoring, and record the evolution of magnitude and phase of the I/Q demodulator output as a function of time.

Due to the high noise level (associated with the large bandwidth) of the AD8302 demodulator, this coarse design has been improved by adding a manual phase-detection scheme – based on a Mini Circuits SYPD-2 – with programmable, high-gain low-noise operational amplifiers for signal shaping. We observe that this low-bandwidth circuit exhibits a noise level low enough for the signal to noise ratio not to be dependent upon the operational amplifier stage gain, but limited only by the phase velocity fluctuations of the SAW delay line due to variations in its environment. The programmable gain of the amplifiers is set using an analog multiplexer. All setpoint identification and gain control tasks are controled by the same ADuC7026 microcontroler.

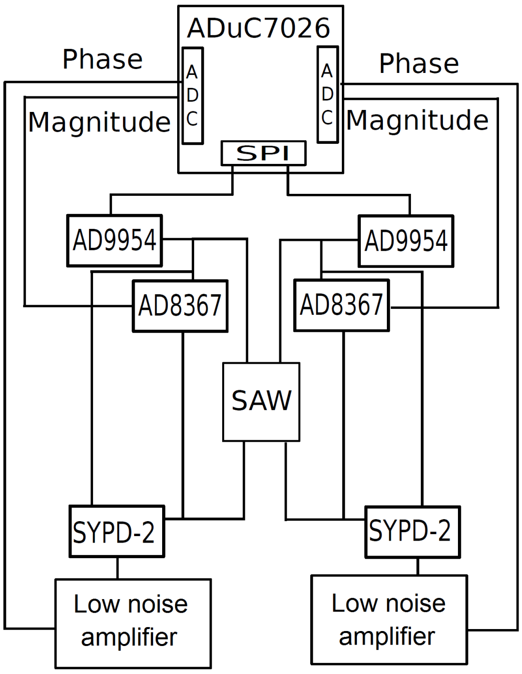


Figure 2: General diagram of the electronic with high accuracy.

The main issue with increasing the gain after the low-noise phase-detection circuit is the limited working range: since the 0-180^o no longer fits in the analog-to-digital converter voltage range, saturation will occur for large mass shifts associated with large acoustic velocity variations. Hence, a phase tracking and unwrapping strategy is implemented, as will be discussed later.

2.2 S_{21} parameter measurement

2.2.1 Coarse measurement

For the preliminary characterization of the delay line, the AD8302 I/Q demodulator provides a mangitude output (29 mV/dB) and a phase output (10 mV/^{o}), the latter with poor linearity close to the measurement boundaries (0 and 180^o). The frequency selection strategy focuses on selecting a setpoint at which the reference and measurement signals are in quadrature, yielding the best dynamics and linearity of the output voltage with respect to the phase between the input reference and measurement signals. Fig. 3 exhibits a typical S_{21} characterization of a Love-mode SAW delay line, in the 118 to 128 MHz range.

2.2.2 Improved accuracy

In order to improve the resolution, a low-bandwidth phase detector is associated with low-noise amplifiers. The phase detector provides a low voltage $v.s$ phase coefficient – 8 mV/degree – but with a noise level low enough to be magnified by an amplification stage. Using variable gain amplifiers, we continuously characterize the SAW delay line phase behaviour as a function of frequency with gains from 1 to 186: switching from one gain to another is controled by the ADuC7026 microcontroler through an analog switch. Fig. 4 displays the evolution of the transfer function with increasing gain. We observe that the phase measurement noise level is independant of the gain (Fig. 6): we attribute the noise level solely to the analog-to-digital conversion step, either due to

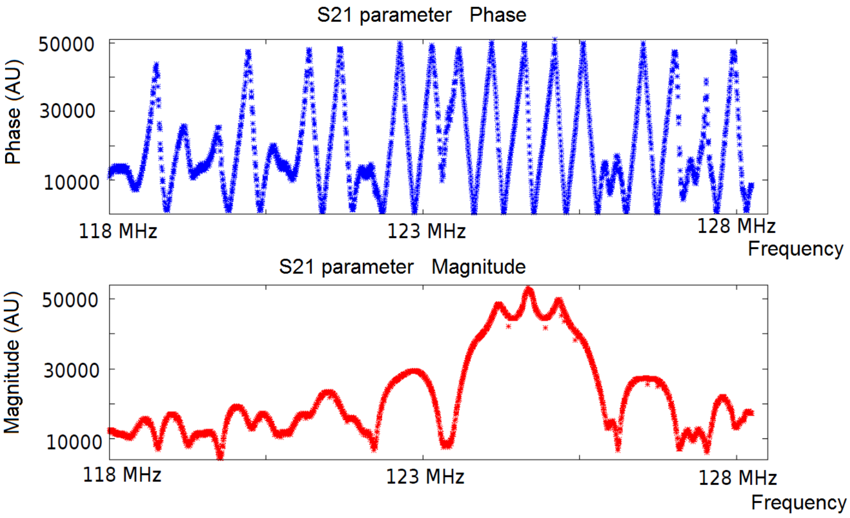


Figure 3: Magnitude (bottom) and phase (top) S_{21} characterization of a Love mode SAW delay line. As opposed to a network analyzer characterization providing a phase measurement in the 0-360^o range, notice the lack of sign information in the phase output of the AD8302, yielding a measurement in the 0-180^o range.

the poor internal voltage reference used by the conversion stage, or inappropriate board layout in separating the analog and digital parts of the power supplies.

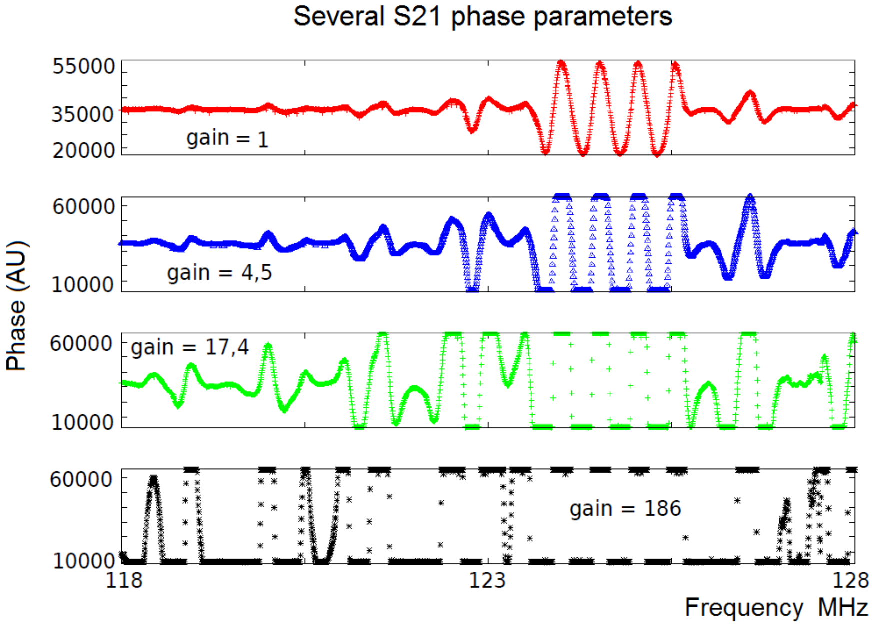


Figure 4: High resolution S_{21} phase characterization of a Love-mode SAW delay line.

This method, which focuses on the phase measurement around the the analog-to-digital converter mid-range, will yield to saturation if the phase shifts during the reaction, a condition which is quickly met at the highest gains. A first strategy is to continuously track the phase, and control the frequency in order to keep the phase close to a setpoint near mid-range. A second strategy requires a preliminary calibration of the phase to frequency

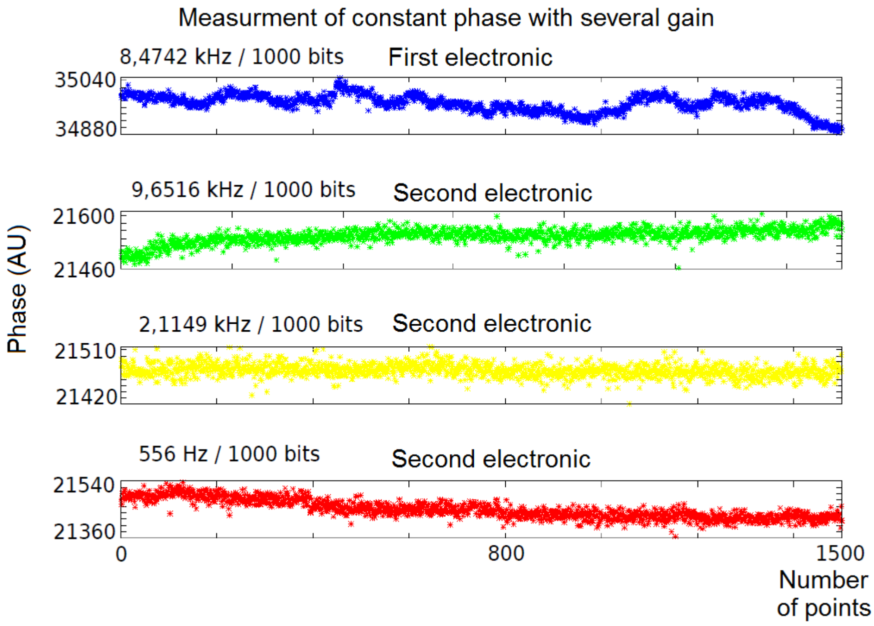
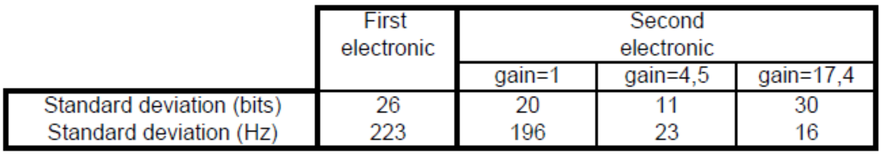


Figure 5: Time evolution of the phase at a constant frequency in order to characterize the noise level as a function of the gain of the low-frequency operational-amplifier based signal processing circuit.



	First electronic	Second electronic		
		gain=1	gain=4,5	gain=17,4
Standard deviation (bits)	26	20	11	30
Standard deviation (Hz)	223	196	23	16

Figure 6: Tabular wich includes standard-deviation in bits and Hz for differents low noise amplifier gains

slope, allow the phase to slowly drift towards one of the range boundaries, and when the boundary is reached, control the frequency in order to bring the measurement close to mid-range.

The former method proved effective for slowly varying signals, with the recording of the frequency as the measured quantity and the phase only providing an error signal as a control of the efficiency of the digital feedback control loop. However, the algorithm which requires continuously reprogramming the DDS with new frequencies is too slow to track quickly varying phase shifts, as will be shown later (section 2.4).

The latter method only requires reprogramming the DDS when the phase signal comes close to the analog-to-digital conversion boundaries. The phase-frequency slope is characterized for each new transducer, and is solely a function of the acoustic wave velocity, which hardly changes for a given kind of acoustic wave (Rayleigh or Love) during a sensing experiment. Furthermore, this frequency-phase slope is dependent on the gain: an array with defined slopes is stored in the memory of the microcontroller for each kind of transducer and for each gain.

2.3 Noise level comparisons

During the coarse qualification step, the typical noise level is of 26-bit units while sampling on a 16-bit range. This noise level is converted to a frequency noise level of 223 Hz, at least two orders of magnitude worst than typical closed loop oscillators noise level. The improved electronics exhibits a constant noise level, independent of the gain (Fig. 5): hence, the relative noise level decreases as the gain increases. The resulting frequency noise level is a standard deviation of 16 Hz. This noise level is still worse than oscillator strategies, but since it is independent of the operational amplifier gain and attributed to the analog to digital conversion noise, faster sampling followed by digital averaging and improved analog circuit layout provide paths towards improving this result.

2.4 Electrodeposition reaction monitoring

Copper electrodeposition has been used for calibrating the gravimetric sensitivity of acoustic sensors thanks to its ability to provide an independent estimate of the deposited metal mass through the measurement of the current generated by the potentiostat [8]. When using a closed loop strategy in order to keep the phase measurement within measurement range of the analog to digital converter at higher gain, the fast reaction rate and large phase shift provide a challenging measurement condition for validating the phase tracking algorithms.

The generation of the voltage driving the potentiostat and electrodeposition parameters (counter electrode voltage and current) are controlled and monitored from the same microcontroller running the SAW monitoring circuits, yielding data synchonization. A typical experiment cycle is exhibited in Fig. 7, with a negative current indicating copper reduction (deposition on the working electrode) and a positive current indicating oxydation (copper removal from the working electrode). Reversibility has been a selection criterion for this experiment since it provides the means of quickly testing various acoustic phase measurement and tracking algorithm parameters: simultaneous to the current monitoring, the acoustic phase and magnitude at fixed frequency are recorded (Fig. 8). At larger gains, as used when recording the data displayed in Fig. 8, the feedback loop on the emitted frequency to keep the phase within measurement range of the analog to digital converter induces a phase wrapping.

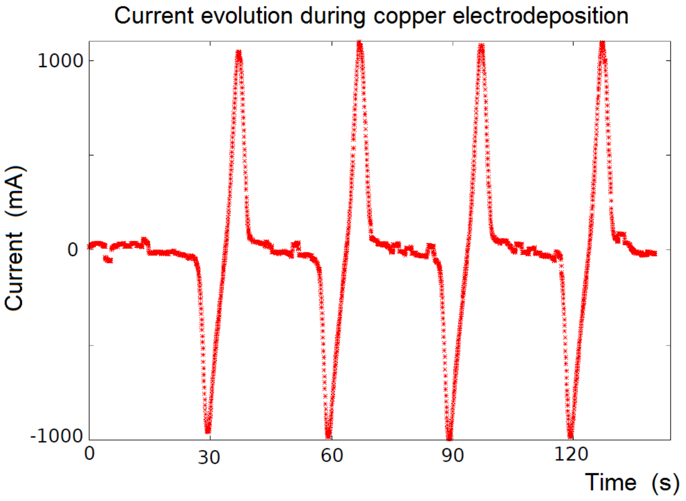


Figure 7: Current monitored during copper electrodeposition reactions.

High resolution phase reconstruction is obtained by unwrapping as displayed in Fig. 8. As can be seen on this figure, the original 16-bit resolution has now been increased to a 17-bit resolution using the given gain: at maximum gain, an additional 4 bits are added to the practical result wih no significant noise level increase.

This experiment demonstrates our ability to monitor the phase of the acoustic signal with high resolution, even for fast chemical reactions. The resulting mass sensitivity of the SAW acoustic sensor, which requires both accurate acoustic phase and potentiostat current measurements, are consistent with previous estimates: values in the 180±20 cm^2/g are consistent with acoustic velocity gradient with respect to the guiding layer thickness (from modelling [9]) and previous experimental estimates [8].

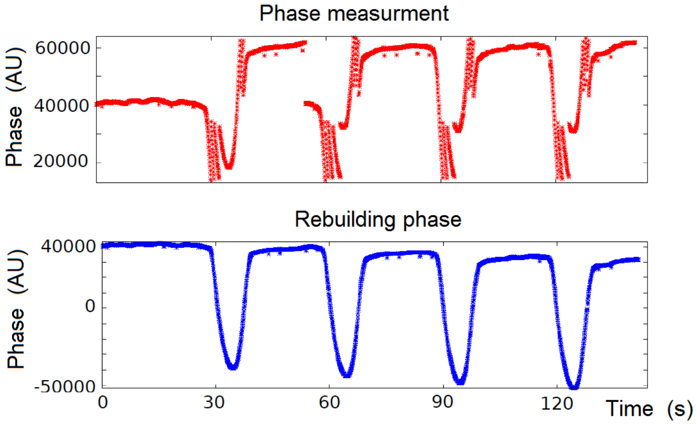


Figure 8: Rebuilding the phase information by unwrapping during copper deposition.

3 Conclusion

We have demonstrated an improvement by a factor 16 of the phase measurement resolution with respect to commercial I/Q demodulators providing a phase output in the 125-434 MHz range. The resolution gain is due to the reduced bandwidth, which is nevertheless sufficient for most chemical reaction monitoring at a few hertz sampling rate at most. This embedded frequency sweep network analyzer combines the robustness of an openloop strategy for the diagnostics of a sensor with poor performances, and the resolution needed for practical gas detection applications.

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