Thermally Modulated Solidly Mounted Resonators for Air Quality Monitoring

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Declaration

I hereby declare that this thesis is submitted to the University of Warwick in support of my application for the degree of Doctor of Philosophy. It has not previously been submitted for any degree application at any other University. Apart from where reference is made in the thesis and the acknowledgement, this thesis is entirely my own work and contains nothing which is the result of collaboration with others.

Parts of this thesis have been published by the author:

Journal Papers:


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Abbreviations
AC – Alternating Current
ACE-2 – Angiotensin-Converting Enzyme 2
AIN – Aluminium Nitride
AQS – Air Quality System
BAM – Beta Attenuation Monitor
BAW – Bulk Acoustic Wave
CMOS – Complementary Metal Oxide Semiconductor
DC – Direct Current
EPA – Environmental Protection Agency
FBAR – Film Bulk Acoustic Resonator
FEM – Federal Equivalent Method
FRM – Federal Reference Method
HEPA – High-Efficiency Particulate Air (Filter)
IDT – Interdigitated Transducers
MCERTS – Monitoring Certification Scheme
MEMS – Microelectromechanical System
MCL – Materials Centre Leoben
OPC – Optical Particle Counter
PC – Personal Computer
PCB – Printed Circuit Board
PLL – Phase-Locked Loop
PM – Particulate Matter
QCM – Quartz Crystal Microbalance
SARS-COV2 – Severe Acute Respiratory Syndrome Coronavirus 2
SAW – Surface Acoustic Wave
SMR – Solidly Mounted Resonator
TCR – Temperature Coefficient of Resistance
TCF – Temperature Coefficient of Frequency
TEOM – Tapered Element Oscillating Microbalance
UFP – Ultrafine Particles
UK – United Kingdom
US – United States (of America)
UHF – Ultra High Frequency
VHF – Very High Frequency
VNA – Virtual Network Analyser
WHO – World Health Organisation
ZnO – Zinc Oxide
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Abstract

The effect of air pollution on the environment and human health is a cause of major concern. Each year millions of deaths are attributed to poor air quality, and it is estimated that its economic cost runs into the trillions of pounds. Especially the pollutant particulate matter has been identified as one of the main contributors to poor health. Hence there is much activity that attempts to reduce the concentration of small particles in air. To better understand the effect of particulate matter on the world and for the effective mitigation of the problems it causes and exacerbates, it is necessary to acquire reliable air quality data. Readily available particle sensing equipment is thus required to expand existing air quality monitoring systems that can deliver meaningful results.

To this end, a range of particle sensing technologies have been studied. Resonator particle sensors based on microelectromechanical systems are one promising example of this because of their potential to provide an affordable solution that can be mass manufactured and use very little power or space compared to many currently available particle monitoring devices. In this thesis a novel particle sensor based on a solidly mounted resonator with an integrated microheater that is compatible with a standard integrated circuit fabrication process is developed and tested experimentally. The main objective of this work is to demonstrate for the first time that temperature modulation applied to a solidly mounted resonator could increase its sensitivity to particles, while targeted particle deposition could increase the effective sensitivity of the system to aerosolised particles and that the application of both could thus help to make this type of sensor more suitable for real world air quality monitoring applications.

The design of the sensor is based upon a complementary metal oxide semiconductor process that includes the deposition of a piezoelectric bulk acoustic wave resonator on top of the standard layer stack. It is verified in an extensive set of simulations and the fabricated sensor is subsequently characterised. In the characterisation study the resonator had a resonant frequency around 2 GHz and a Q factor of approximately 200. The device was found to be capable of handling temperatures induced through the application of an electric current to the integrated microheater of up to 598 K. Experimentally the device’s resonant frequency, S-parameter value and its temperature for different applied currents were found to be within approximately 6% of the sensor simulations.

A custom particle test rig was built to evaluate the sensors performance as a particle sensor. One of the main obstacles remaining with these types of sensors is the reliability of particle measurements, which is reduced by difficulties to achieve repeatable particle sampling. To resolve...
this issue a thermophoretic particle deposition channel was added to a commercial FBAR device and experimental tests were carried out that showed it could reduce the variation in measurement results between repeat tests from 71% to 14%.

The novel solidly mounted resonator particle sensor device was tested inside the particle test rig and found to have a sensitivity to particle deposition of approximately 40 Hz/ng. Temperature modulation was applied to the sensor through the integrated microheater and this was found to increase the sensitivity of the device by a factor of almost five to 190 Hz/ng. It also reduced the sensor’s detection limit from approximately 100 ng to 50 ng. The thermophoretic microchannel was added and found to approximately double the sensitivity of the novel sensor to airborne particles through increased particle sampling efficiency. The novel thermally modulated SMR particle sensor was found to have significant potential for low-cost quality monitoring applications.
1 Introduction

1.1 Air Quality Monitoring

Poor air quality is one of the leading causes of deaths worldwide and this is increasing [1], [2]. For example, in 2012 the World Health Organisation accounted approximately 3 million deaths to its effects on human health [3], [4], while in 2016 it attributed an estimated 7 million deaths to the effects of air pollution and the resulting poor air quality indoors and outdoors [5]. In 2016 deaths from air pollution were estimated to be responsible for a global economic cost of US$4.1 trillion [6]. It is expected that the cost of air pollution to the public health sector of the United Kingdom will rise to £5.1 billion (about US$7 billion) by 2035 [4], [7]. Air pollution is projected to increase in the next years, with some scenarios suggesting there could be a doubling of the effect air pollution has on premature mortality by 2050 [2], [3], [8].

To mitigate the impact of air pollution on human health more extensive, more detailed, and more accessible pollution data is required, which necessitates the development of accurate, low-cost monitoring equipment [9]. Microelectromechanical system (MEMS)-based sensors have strong potential to provide a solution in this field of research [4], [10]–[18]. Hence, the aim of this research is the development of a novel low-cost particle sensor based on complementary metal oxide semiconductor (CMOS) technology. Furthermore, methods aiming to improve its performance through particle sampling based on thermophoresis and increased sensitivity based on modulation of the sensor’s temperature are investigated.

1.1.1 Particulate Matter

Particulate matter is considered the airborne pollutant with the strongest effect on human health [19], [20]. It is particularly a problem in large, densely populated cities, especially in developing countries [3]. Human populations exposed to particulate matter over the long-term show increased rates of mortality and cardiovascular incidents compared to unaffected populations [20]. It was also found that exposure to particulate matter exacerbates respiratory diseases [20]. The United States Environmental Protection Agency (US EPA) also concludes that there is causality between human cardiovascular effects and mortality and exposure to particulate matter pollution [21]. Especially particulate matter smaller than 2.5 µm in diameter (PM2.5), which includes ultrafine particles (UFPs) with a diameter under 0.1 µm, has a negative effect on human vulnerability to lung cancer, cerebrovascular and cardiovascular functions, and the central nervous system [1], [22].
More recently, rates of COVID-19 mortality have been attributed partially to air pollution, because of a potentially exacerbating effect of particulate matter on disease symptoms [18], [23]. Since the enzyme angiotensin-converting enzyme 2 (ACE2) is considered to serve as a receptor for Sars-CoV-2 in humans [23]–[25], the increased expression of ACE2 in humans exposed to particulate matter might partly explain the relationship between deaths from COVID-19 and regional air pollution as shown in studies conducted in [23]–[25]. It is further thought that pollution has had a contributory effect to regional variations in infection rate and hence case numbers [26]–[28]. While the main reason for the correlation between air pollution and case numbers is most likely the causal relationship between population density, as well as temperature, and case numbers [29], [30], it is still thought that airborne particulate matter can act as a transport agent for the SARS-CoV-2 RNA [31]–[33]. An example in support of this theory is that it has also been considered for influenza in the past; the studies conducted in [34]–[36] postulate that there is a connection between peak pollution levels and peak influenza infection levels [34]–[36].

Particulate matter emission sources are both anthropogenic and natural [18]. Anthropogenic sources include combustion engines, energy production and other industry, household consumption and agriculture, while the most common natural examples are erosion and desert dust [1]. Example constituents of particulate matter include particles of black carbon, dust from erosion, sulphate, and pollen [18], [21]. In Europe agriculture is the largest contributor to particulate matter pollution, followed by transport [2]. Particulate matter is usually classified by diameter; of most interest are sizes up to 2.5 µm (PM2.5) and up to 10 µm (PM10) [18], [21].

1.1.2 Need for Particulate Matter Monitoring
Regulations aiming to lead to reduced particulate matter concentration exposure are in place in many countries [18]; governments usually define a maximum average particle concentration over a certain timespan in a certain space [1], [8], [21], [37]. The World Health Organisation currently recommends a limit of 10 µg/m³ of PM2.5 in its Air Quality Guidelines [8]. In the US the 24-hour PM2.5 limit is currently 35 µg/m³ and the annual maximum is 12 µg/m³ (October 2021) [21]. Compared to other air quality indices, pollution standards imposed over smaller timescales and smaller areas have been found to lead to increased health benefits, but the availability of localised, real-time pollution data currently reduces the effect of these regulations [4], [18], [21], [38], [39].

Time averaging of pollution data is currently used to determine the average air pollution over specific time intervals, which are defined in many air quality indices. In the United States of
America, the current averaging time is 24 hours, for example [21]. A consequence of this is the dependence of data reliability on the length of the discrete sampling intervals the deployed pollution monitoring equipment can achieve [38], [39]. For accurate averaging, devices need to be able to provide very frequent, real-time data or be capable of capturing and holding particles for a very long time between measurements. A straightforward way of reducing the impact of air pollution on human health is the avoidance of affected areas by individuals of concern [10], [40]–[42]. This is hindered by the use of long measurement intervals, as it often leads to a smoothing effect, ignoring the instantaneous spikes in pollution at-risk populations should avoid [18], [38]–[40].

This is also true for spatial variations of PM concentrations [18], [42]. The reliability of regional pollution data is dependent on the spread of PM monitors, of which there is a limited number, in a space of concern [38], [39], [42], [43]. For example, if multiple PM monitors are placed apart from each other there is no way to know the exact concentration at all points between the monitors. For this reason, pollution data is often averaged spatially [39], [44]–[46]. This gives rise to data inaccuracies, especially in areas with high local variations in air quality [38], [39], [46].

In a review of the current PM air quality standards, the United States Environmental Protection Agency (US EPA) points out the effect of spatial averaging on pollution data, especially its “disproportionate impacts in at-risk populations, including minority populations and populations with lower socioeconomic status”, because of the generally observed poorer air quality in locations with more deprived populations [21]. Spatial averaging has thus been abandoned for US EPA pollution policy reviews, for example [21]. Possible solutions for better pollution data include the use of mobile monitoring stations, although these will be less accurate when data is time-averaged [47]. It is thus also desirable to increase the number of monitoring stations for more accurate measurements, be they mobile or stationary [38].

A widespread PM monitoring network, or even personal monitoring devices, can thus improve spatiotemporal pollution data coverage and help individuals to avoid poor air quality more effectively [10], [38], [39], [41], [48]. To realise this, it is necessary to advance readily available, accurate, low-power, low-cost instrumentation equipment for real-time particulate matter measurement [4], [9], [10], [18], [38], [39], [41]–[43], [49], [50].

1.2 Thesis Outline
The remainder of this thesis is structured as follows:
The first part of **Chapter 2 Background and Literature Review** discusses the most widespread particulate matter monitoring methods. The working principle of each method is discussed together with advantages and disadvantages of each method and lists some commercial examples. Recent attempts to make low-cost MEMS particle sensors are also discussed. Since this research focuses on MEMS sensors, the second part of Chapter 2 outlines methods of particle sampling and separation that can be used to improve MEMS particle sensors.

**Chapter 3 Solidly Mounted Resonators** explains some relevant theoretical background of particle sensors based on piezoelectric resonators, with particular focus on solidly mounted resonators and the circuitry required to use them in sensing applications.

The design and simulation, as well as the practical characterisation of a novel solidly mounted resonator particle sensor with an integrated microheater that is based on a standard integrated circuit manufacturing process, is presented in **Chapter 4 Design and Fabrication of a Thermally Modulated CMOS SMR**. It discusses some of the trade-offs made in the design stage of the sensor based on simulation results and shows a functional verification of the fabricated device in terms of resonant and thermal performance.

The first part of **Chapter 5 Thermophoretic Particle Deposition** explains the design and build of a test rig for particle sensors. Its central elements are a glass test chamber and an aerosol generator, as well as a computer control interface. The second part of Chapter 5 presents the results of practical tests of a piezoelectric resonator device as a particle sensor that were carried out with this test rig. The sensor was combined with a thermophoretic particle deposition device to increase its sensing performance.

**Chapter 6 Thermally Modulated CMOS SMR for Particle Sensing** presents practical particle sensing tests of the novel solidly mounted resonator demonstrated in Chapter 4. Chapter 6 present proof-of-concept tests of a novel thermal modulation technique to increase the sensitivity of the solidly mounted resonator to particles.

**Chapter 7 Conclusions and Future Work** summarises this thesis and suggests some possibilities for future work on solidly mounted resonator particle sensors.
2 Background and Literature Review

2.1 Measurement of Particulate Matter Concentration

Instruments for the measurement of particle concentration usually focus on measuring either the surface area the particles take up, their combined mass or the number of particles in a certain volume [51]. Important indicators of performance of any sensor are their response time (or achievable sampling rate), specificity (ability to detect only the desired quantity/particle size), detection limit (minimum measurable quantity or mass of particles) and detection range, as well as sensitivity (magnitude of response per unit change in quantity) and resolution [52]. Different methods are used to measure particle concentration, including charge based, optical or weight-based methods [18], [51]. Many countries draw on a specific set of approved particulate matter monitoring equipment. For example, the US EPA Air Quality System (AQS) currently classifies approved measurement devices for PM2.5 or PM10 concentration into Federal Reference Methods (FRMs) and Federal Equivalent Methods (FEMs) [21]. FRMs and FEMs approved by the US EPA use gravimetric methods, beta attenuation monitors or optical particle monitoring equipment [18], [53], [54]. The United Kingdom approves similar methods through the Environment Agency using the Monitoring Certification Scheme (MCERTS) [55]. Its list has significant overlap between devices approved in the US and the UK [54], [56]. Both FRMs and FEMs are generally expensive devices, with FRM costs potentially running into tens of thousands of dollars [57]. The US EPA designates devices costing under $2500 as low-cost sensors, neither of these are currently classified as FRMs nor FEMs [54], [58].

2.1.1 Approved Gravimetric Methods

Gravimetric methods using a particle sampler make up most of the US EPA approved FRMs. The polluted air is passed through a filter by a sampling fan, the filter then accumulates the particles over time. The total change mass of the filter over the sampling time and the volumetric air flow rate is then used to calculate the particle concentration [18], [50]. A schematic of a particle sampler is shown in Fig. 2.1.
Particle filter-based PM monitors suffer from less dependence on sampling frequency when used to establish values of average PM pollution over long time intervals, which, on the one hand, makes them suitable to monitor, for example, the US EPA’s 24-hour standards reliably. On the other hand, the need for periodic particle filter weighing makes the collection of real-time data, and hence short surges in pollution levels, difficult with these types of devices [18], [59], [60]. Samplers also require multiple filter stages to establish particle size distribution. Impactors or other sorting mechanisms are often used to remove large particles [51]. Cascading impactors can improve classification of particle size distribution [51]. The FRM approved BGI PQ200 Ambient Air Particulate Sampler is about 90 L in volume and weighs about 27 kg, with a 32 W solar panel power supply. It has a 24-hour sampling interval for PM10 and PM2.5 [61]. Other examples of commercial FRM particle samplers include the Met One E-SEQ-FRM [62] and the Partisol™ 2000i Air Sampler by Thermo Scientific™, shown in Fig. 2.2 [63]. Partisol™ series devices are also approved in the United Kingdom [56]. They have similar volume and weight to the BGI PQ200, but require a permanent power supply of around 300 W.

Fig. 2.1 Particle Sampler Schematic.
Because of the need for a significant amount of manual work on particle samplers and thus on nearly all FRMs, the US EPA now increasingly uses automated FEMs [21], [54].

Currently the only automated, approved, and gravimetric FEMs are tapered element oscillating microbalances (TEOM) [54]. A filter collects particles at the end of a hollow, oscillating channel [64]. The accumulation of particles on the filter changes its mass and thus shifts its oscillation frequency, which is measured by a frequency counter [18], [59], [60]. A schematic of this is shown in Fig. 2.3.
This automated read-out mechanism also enables continuous measurements [18], [21]. The only currently approved FEM and UK MCERTS certified TEOMs are the 1405 TEOM™ Continuous Ambient Particulate Monitor and the Continuous Ambient Particulate TEOM™ Monitor Series 1400ab by Thermo Scientific™ that is shown in Fig. 2.4 [54], [56].

**Fig. 2.3. Tapered Element Oscillating Microbalance (TEOM) Schematic.**
Both weigh 18 kg excluding control setup and outdoor enclosure, have a comparable volume to the particle samplers, but 600 W power consumption [65], [66]. Their inlets require configuration specific to the desired PM diameter. At 0.1 µg/m³ resolution and an accuracy better than ± 2.0 µg/m³, they can measure up to 1 g/m³ and 5 g/m³ of selected PM respectively [65], [66].

A drawback of both particle samplers and TEOMs is their need for regular filter maintenance [18], [60]. Both devices also struggle with fluctuating relative humidity [18]. The air sample is commonly heated before it reaches the collection filter to overcome this by evaporating droplets of vapour [18]. Sample heating makes the detection of volatile particulates difficult [64].

2.1.2 Beta Attenuation Monitoring
Beta attenuation monitors (BAMs) are automated FRMs and FEMs for particle monitoring [9], [21], [54], [67]. BAMs comprise a source of Beta radiation, as well as a Geiger-Mueller counter [18]. They use a filter belt to collect particles and measure the resulting attenuation of the beta rays directed at the filter from the source [18], [67]. A schematic of a beta attenuation monitor is shown in Fig. 2.5.
A BAM delivers particle concentration data at higher frequencies than current FRM and FEM gravimetric methods [18]. However, they also suffer from humidity dependence, resulting in inaccuracies and air drying [18], [67]. The first FEM approved BAM was the Met One BAM 1020, which is also used in the United Kingdom and makes up more than half of the US EPA’s deployed, continuous FEMs [21], [56]. It has a resolution of 0.1 µg/m³ and an accuracy better than ± 2.4 µg/m³. When equipped with appropriate particle selection accessories, the BAM 1020 can automatically measure PM10 and PM2.5 up to 10 mg/m³. It is shown in Fig. 2.6 [68]. With a weight of 19 kg, and volume of 63 L it is comparable in size to the other FRM methods [68]. The power consumption of the BAM 1020 typically ranges from 262 W to 848 W depending on the configuration [68].
2.1.3 Optical Particle Monitoring

Most optical particle monitors are fully automated, continuous devices and measure the scattering of light from particles targeted by a laser beam and hence count their number [18], [50], [69], [70]. This type of device is also called an optical particle counter (OPC) [18], see the schematic in Fig. 2.7.

Fig. 2.6. *Met One BAM 1020* [68].
The main downside of optical particle counters is the need for calibration for different particle diameters and even materials [18], [69], [71]. However, as opposed to the filter-based monitoring methods outlined above, once calibrated, measurement of particle size distribution can be achieved in a single device by using separate light channels to count particles of each desired diameter [69]. They can also be used in conjunction with particle separation mechanisms that take this into account, for example as described by Keskinen et al. [72] and Dong et al. [73]. To differentiate between PM and vapour droplets, high-end OPCs also commonly dry the air samples used. Both currently approved optical FEM particle monitors, the Grimm Model EDM 180 PM2.5 Monitor (which is also MCERTS certified in the United Kingdom), shown in Fig. 2.8 [74], and the Teledyne Model T640 PM Mass Monitor, offer this option [74], [75]. They use 116 W and 90W of power respectively. In the case of the GRIMM EDM180, power consumption can be reduced to 18 W when used without the heating element. The volumes of these monitors are about 47 L and 28 L with weights of 18 kg and 11.3 kg. Both approved optical monitors are significantly smaller

![Optical Particle Counter (OPC) Schematic](image-url)

**Fig. 2.7. Optical Particle Counter (OPC) Schematic.**
and even lighter than the other approved methods mentioned here, while achieving the same resolution of 0.1 µg/m³. Their detection limit is 10 mg/m³ for PM10 [74], [75].

Fig. 2.8. Grimm Model EDM 180 PM2.5 Monitor [74].

Non-approved low-cost OPCs are also widely available [17], [18]. They can be found in substantially smaller configurations than the methods mentioned above [18], and some are currently available for under $100. The Honeywell HPM Series OPCs, for example, are available for under $50, smaller than 37 mL and use only 400 mW of power [76], [77]. Another commonly used low-cost PM monitor is the Alphasense OPC-N2 ($300 [58], now superseded by the OPC-N3 [78]). This OPC has a power consumption of about 0.9 W. The main issue with low-cost OPCs is limited correlation between their readings and FEMs. Optical methods generally work better in lower concentrations because of limitations to the achievable counting speed and laser accuracy. Crilley et al. have shown limitations of low-cost optical particle counters when measuring PM1, PM2.5 and PM10 without a correction factor by evaluating an OPC-N2 against established reference devices [79]. The US EPA has also reviewed an OPC-N2 and a Dylos DC1100 ($1000), as well as some other devices costing between $1000 and $2050 and found them to be of limited accuracy (R² values of 0.01 and 0.58) [58], [80]. In this test the best performing was the $2000 RTI MicroPEM with an R² value of 0.71 when compared to the Grimm 180 [58], [80], [81]. The Sensirion SPS30 is another OPC currently available for under $50. It weighs 26 g with a volume of 19 ml and has a power consumption of 275 mW [82]. It is certified by the UK Monitoring Certification Scheme (MCERTS) for the Performance Standards for Indicative Ambient Particulate Monitors. This does not mean it can be used in European air quality networks, but it does mean it
is approved for use for quantitative PM measurements with an uncertainty better than ±50% if each device is calibrated on-site with an approved reference method [82].

Another issue with optical particle sensors, which increases their power consumption, is the need to maintain stable airflow that is sufficient to produce an adequate air sampling rate, but low enough to allow for enough time to detect the particles optically [18], [83]. Particle contamination of OPC sensing elements is another issue pointed out, for example, by Dong et al., who otherwise found a setup involving a commercial optical sensor to be accurate. They also mention the need for frequent sensor calibration and suggest the use of a compensation algorithm to increase sensor accuracy [73]. Gao et al. attempted to counter sensor drift caused by flow variations and time-varying and temperature dependent laser intensity or photodiode sensitivity, by developing a GPS-based adjustment algorithm to account for localised variations on different bus routes [84]. Castello et al. proposed a similar mechanism to improve the accuracy of low-cost particle sensors mounted onto buses in 2021 [85]. The main downside of these approaches is their requirement for extensive data from an extensive grid of ambient monitoring stations and thus significantly increased system complexity and cost. Shao et al. also proposed a software compensation mechanism; however, their RMS value measurement approach requires runtimes more than 120 hours to achieve high accuracy results, which limits the device’s ability to collect real-time data [84]. A further drawback of optical devices is their power consumption since they need to power a sampling mechanism (fan to provide sufficient airflow) and a light source on top of the read-out mechanism. As mentioned above, low-cost OPCs tend to use substantially less power than larger filter samplers, BAMs and TEOM devices [18], [76]. Another drawback is limited compatibility to integrated circuit technology. OPCs cannot take advantage of the low cost of mass manufacturing associated with the latter. This is one reason for their generally high prices [86]. Wang and Muth tried to counter these drawbacks by utilising optical fibre to reduce the length of the optical path between the light source and the particles, reducing the required laser power and system size, but pointed out the need for further investigation when attempting to sense different particle sizes with their optical sensor [86].

2.1.4 Other Particle Monitoring
Many other particle monitoring techniques have been investigated. For example, Hirleman described the Phase-Doppler Particle-Sizing Velocimeter as a crucial development for in-situ determination of both particle size and velocity [87] and in 2015 Morita et al. presented a method
to enable mass-production of these devices [88]. However, these devices have been used mostly for single-particle detection and not necessarily for concentration measurement [89], although the devices can be combined with particle counter technology [90]. In 1999 Zipser and Franke proposed an aerosol sensor based on the acoustic absorption effect. One transducer generates a sound wave at one end of a chamber and other measures the remaining pressure amplitude of the wave [91]. The device is however limited to the sensing of liquid aerosol components and is hence not suitable for solid particulate matter [91]. In 2016 Zhou et al. proposed using a grounded electrode to collect naturally charged particles. The pulses generated by the resulting transfer of electrostatic energy are then measured [92]. Observed currents in the pA range limit the practicality of this sensor for low-cost applications [92].

2.1.5 MEMS Particle Sensors

Another, promising field of particle concentration measurement is the use of microbalances based on microelectromechanical systems (MEMS), manufactured through micromachining processes [18]. Like TEOMs, these gravimetric sensors typically employ resonant piezoelectric (deformation induces a potential difference across the device) or piezoresistive (deformation causes a change in resistance) elements, where the particles accumulate on a resonant body and hence alter its frequency of resonance [18], [51].

The sensitivity, $S$, of resonant particle sensors is given by (2.1), $\Delta f$ is the change in frequency and $\Delta m$ is the change in analyte mass (here particle mass) [52].

$$S = \frac{\Delta f}{\Delta m} \quad (2.1)$$

The sensitivity is commonly divided by the fundamental resonant frequency to scale $S$ for the comparison of sensors with different resonant frequencies [52]. However, probably because practical sensor resolution is determined by the absolute measurable frequency shift rather than the relative one and because the measured shifts in resonant frequency are strongly dependent on the coupling of the sensor and the particles and thus the size and the position of the particles on the sensor surface [93], [94], most of the literature on MEMS particle sensors studied does not do this, as will become clear in the following. Particle position is especially difficult to control when working with airborne particulate matter that deposits on the sensor surface relatively randomly, as in this case a simple frequency readout of the sensor does not contain information on the particle position or size on the sensor surface.
The main reason these devices are used in resonant mode is the generally increasing mass sensitivity with increasing frequency for each type of device [4]. Mass sensitivity is their main limitation thus far [18]. In addition, operation at resonance also gives rise to phase noise, which means frequency shifts need to overcome a certain noise floor to be distinguishable from frequency fluctuations unrelated to the analyte [52], [93]. They have large potential to resolve some of the issues with the sensor technologies mentioned above, especially those regarding instrument size and power consumption, because they are microscale devices [18]. Because of their potential for simple, large-scale manufacturing, MEMS sensors are of particular interest for low-cost monitoring equipment [4], [10]–[18]. Complementary metal oxide semiconductor (CMOS) technology can be used in combination with MEMS fabrication to make fully integrated sensors [94]. The circuitry required for sensor drive and readout could be combined with the sensor itself to make system-in-package, or even system-on-chip particle sensors [18]. Low-cost, large-scale manufacture is thus achievable [4], [12], [17], [18], [95]–[97]. The following will discuss some attempts to make MEMS particle sensors.

Hajjam et al. developed a piezoresistive resonator based on thermo-electric transduction, which means the device is heated and cooled sufficiently fast to bring the cantilever to resonance [98]. One advantage of this mechanism is that it does not require integration of a piezoelectric material and can be manufactured easily, but, because of typical thermal transients achievable in MEMS structures, its frequency is limited to the megahertz range, leading to lower sensitivity [98]. Wasisto et al. demonstrated a silicon MEMS cantilever device (called CANTOR) with a theoretical mass sensitivity of 36.5 Hz/ng, based on a frequency shift observed from airborne particle sampling. This device was resonated by means of external actuation [99]. They removed this requirement in a second version of their device in 2015 [100]. The sensor has a relatively large power requirement for a MEMS sensor (1.25 W), but a portable volume of 540 mL, with a weight of 375 g [100]. Setiono et al. presented an electrothermal MEMS cantilever particle sensor with a sensitivity of 13 Hz/ng in 2020, which is promising, providing sufficient particle sampling efficiency and repeatability can be achieved [101].

Piezoelectric sensors require less power than piezoresistive sensors and can even induce their own signal [102], [103]. Piezoelectric, cantilever-based mass sensors are very common, simple MEMS structures, and they exhibit high mass sensitivity [104]. Piezoelectric cantilevers are typically made to resonate through voltage perturbations at interface electrodes near their resonant
frequency, see Fig. 2.9. For example, in 2019 Joshi et al. achieved a mass sensitivity of 314 Hz/ng using a manually deposited proof mass [105] and in 2020 Fort et al. were able to achieve an experimental sensitivity of 100 Hz/ng (significantly higher than the electrothermal device by Setiono) with a theoretical detection limit of 10 µg [104]. Experimental values have also demonstrated the functionality of piezoelectric cantilever aerosol particle sensors in principle [104].

It is expected that future cantilever particle sensors may deliver better sensitivity results, much of the current work is based on a limited number of tests for particle sensing [100]. This also makes it difficult to compare sensitivities, as repeatability of sampling can be an issue, see section 2.3.1. Bertke et al. calculated a theoretical detection limit improvement to the CANTOR piezoresistive cantilever particle sensor down to 0.26 pg in 2021, achieved mainly by improved sampling [106].

Weng et al. developed a similar sensor in 2020, based on a thin-film piezoelectric-on-silicon MEMS oscillator [107], [108]. The device was demonstrated to be capable of detecting silver droplets with a high sensitivity of approximately 5 Hz/pg [108]. It is likely that this sensitivity would be somewhat lower for PM deposition, since PM particles might have weaker coupling to the resonator than the silver droplets. Weng et al. also deposited the droplets on the most sensitive part of their device, which would be difficult to replicate with the same accuracy when attempting to capture aerosolised particles [4], [94], [107], [109].

A particle sensor based on a resonating AlN film with platinum/titanium electrodes on a SiN/SiO$_2$ membrane was developed by Choi et al. in 2020 [110]. The device has a fundamental resonant frequency of 125 kHz. After manual particle deposition the authors were able to observe
a particle sensitivity of up to 354 Hz/ng at a harmonic of the resonant frequency around 2 MHz [110].

2.1.6 Acoustic Wave MEMS Particle Sensors

The most common microbalance besides TEOMs is the quartz crystal microbalance (QCM) [51]. It is made up of a piezoelectric quartz crystal with electrodes on either side, see Fig. 2.10. Sauerbrey found quartz crystal resonator mass loading to be proportional to the change in resonant frequency of the resonator in 1959 [111]. The accuracy of his device was found to be ±1 ng [111]. The particles are accumulated directly on its surface through particle precipitation or adhesion and thus induced change in resonant frequency is measured from the resulting change in piezoelectric layer thickness [18].

![Fig. 2.10. Quartz Crystal Microbalance (QCM) Schematic.](image)

One advantage of QCMs is their low achievable temperature coefficients of frequency [112]; a major problem on many other resonant mass sensors [10], [52], [113]. Currently one of the main drawbacks of QCMs for particle sensing is their low particle mass sensitivity, caused by constraints to achievable crystal thicknesses that limit their resonant frequencies [18], [114], [115]. One way to solve this problem could be to reduce the number of particles that do not bond to the resonator surface and thus achieve greater mass deposition on the resonator surface for a given concentration of aerosolised particles. This would increase the sensitivity of the sensor to aerosolised particle concentration without increasing the actual mass sensitivity of the device [112]. By printing a grid of vertical micropillars onto a QCM surface, Hajizadehmotlagh et al. were able to demonstrate an improvement in particle capture efficiency of about 25%, resulting in improved QCM particle sensor performance [112], [116].

Other sensors based on surface and bulk acoustic wave devices (SAWs and BAWs), very similar to the QCM in Fig. 2.10, have been demonstrated for particle sensing in previous work [4], [10]–[13], [15]–[18], [70], [117]. Acoustic wave sensors are MEMS sensors based on either
specially cut piezoelectric crystals or piezoelectric thin-film technology using materials such as zinc oxide (ZnO) or aluminium nitride (AlN) [4], [12], [18], [94], [117], [118]. The latter technology enables significant increases in resonant frequencies and thus, potentially, sensitivities [99], [119]. Surface acoustic wave devices are made by manufacturing interdigitated transducer electrodes on the surface of a piezoelectric substrate [117]. The spacing of the transducers is proportional to the wavelength of the acoustic wave. A sinusoidal voltage applied across metal electrodes excites the standing acoustic wave in the piezoelectric film. Particles on the surface of the device increase not only the sensor’s mass, but also its thickness. The wavelength of the excited acoustic wave is increased this way and thus high sensitivity to particle deposition, as well as particle discrimination is theoretically possible [4], [94]. Section 3 discusses the working principle of acoustic wave resonators in more detail.

In 2013 Thomas et al. described a 262 MHz quartz SAW sensor for 750 nm gold particles, with sensitivity of 175 Hz/ng, which could also be used for particulate matter monitoring [115]. A 262 MHz SAW sensor was also shown by Thomas et al. to be sensitive to 10, 8, 4 and 2µm particles with a sensitivity of 275 Hz/ng in 2016 [117]. These devices were tested with manually deposited rather than aerosolised particulate matter. Liu et al. successfully detected particulate matter from aerosol with a 312 MHz SAW device in 2017 [120].

Although SAW sensors are easier to manufacture and have higher mass sensitivity than BAW sensors, the achievable resonant frequencies of BAW sensors are now substantially higher [12], [13]. This makes them an interesting alternative to SAW devices for particle sensing. Additionally, BAW sensors also exhibit lower power consumption than those based on SAW technology [119]. A BAW device is manufactured by depositing a piezoelectric thin film between two (top and bottom) electrodes [18]. The resonant frequency and hence the sensitivity is mainly dependent on film thickness [18], [94]. For optimal performance, an air cavity is etched out underneath the sensor, this type of device is called a film bulk acoustic resonator (FBAR) [4], [18], [52]. The air on both sides of the sensor leads to the optimal trapping of energy inside the thin film. In 2010 Paprotny et al. designed an FBAR sensor for particulate matter monitoring for PM2.5, with an expected sensitivity of a few µg/m³ [121]. Section 2.3.1 discusses this device in more detail.

Solidly mounted resonators (SMRs) are BAW devices where the air cavity is replaced with a Bragg reflector stack [18]. In 2014 Villa-Lopéz et al. presented a ZnO design of a SMR for particle sensing with a resonant frequency of 875 MHz [122]. They estimated a theoretical
sensitivity of about 5 kHz/pg for PM2.5 and 250 kHz/ng for PM10 [15], [122]. The physical implementation of this sensor was tested in 2016 and detected deposited particles successfully [118]. One limitation to the resonator in this paper is a manufacturing error of up to 21 % in the thickness of the piezoelectric layer [118], which obstructs the assessment of mass measurement accuracy. Thomas et al. observed this 875 MHz SMR to have a sensitivity of 4.6 Hz per µg/m³ [12]. A 1.5 GHz ZnO SMR was also designed by Villa-Lopéz et al. [118]. Simulations of this sensor resulted in a substantially larger sensitivity to PM2.5 than that of the 875 MHz device [94]. This highlights the effect of increasing resonant frequencies.

SMRs have been found to be less responsive to mass loading than FBARs [123]. The removal of the airgap increases device ruggedness and decreases sensitivity to temperature and pressure [94]. Additionally, FBARs lack the ability to easily add layers for temperature compensation or control, since the airgap insulates them from the substrate. Furthermore, FBARs are more difficult to manufacture than SMRs because of the need to etch an airgap into the device. This also reduces compatibility with many integrated circuit processes [18]. A limitation to SMR devices manufactured completely in processes used for CMOS manufacture is the design of the Bragg reflector [124]. Processes typically have standardised materials and thicknesses of their metal layers [18], [95], [125]. For example, aluminium might be used rather than tungsten or molybdenum, which does not give the material stack an optimal acoustic impedance ratio between the high and low acoustic impedance layers [18], [94], [125]. Research must show to what extent this limits the mass sensitivity of such sensors. It is also possible that combined CMOS-BAW processes could offer high acoustic impedance metal layers for the Bragg reflector in the future.

Edrees et al. manufactured a 1.75 GHz ZnO SMR on top of a 65 nm CMOS Pierce oscillator circuit in 2017 and demonstrated the functionality of their very compact system [124]. They achieved a quality factor of 460, which was reduced by the addition of the CMOS oscillator from a SMR Q-factor of 500 on glass. Villa-Lopéz et al. deposited an AlN BAW device onto a Bragg reflector manufactured in a standard CMOS process and demonstrated its ability to detect deposited particles [95]. Both works required post-processing beyond the process available for mass manufacture.

2.1.7 Table of Measurement Methods
To summarise, the most important points from section 2.2 are presented in Table 2.1 [18].
Table 2.1 Particle Monitoring Methods [18].

<table>
<thead>
<tr>
<th>Method</th>
<th>Working Principle</th>
<th>Advantages</th>
<th>Drawbacks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle Sampler (gravimetric)</td>
<td>Fan passes polluted air through a filter that captures the particles [50]. Particle concentration is calculated from volumetric air flow rate and the change in mass of the particle filter</td>
<td>- Accurate for long measurement intervals [54]</td>
<td>- Expensive [57] &lt;br&gt; - Required filter weighing and cleaning [59], [60], [126] &lt;br&gt; - High power consumption [57] &lt;br&gt; - Bulky [53], [54]</td>
</tr>
<tr>
<td>Tapered Element Oscillating Microbalance (TEOM) (gravimetric)</td>
<td>Hollow, oscillating channel with a particle collection filter at its end [64]. Frequency counter records shift in oscillation frequency caused by a change in collected particle mass [59], [60].</td>
<td>- Can provide real-time data [21] &lt;br&gt; - Widely used [53], [54]</td>
<td>- Expensive &lt;br&gt; - High power consumption &lt;br&gt; - Need filter cleaning or replacement [60] &lt;br&gt; - Humidity dependence can lead to inaccuracies, requires air heating [64] &lt;br&gt; - Bulky [53], [54]</td>
</tr>
<tr>
<td>Beta Attenuation Monitor (BAM)</td>
<td>Measure the attenuation of beta rays (i.e., electrons) by particles on a filter with a Beta radiation source and a Geiger-Mueller counter [67]</td>
<td>- Can provide real-time data &lt;br&gt; - Widely used [9], [21], [54], [67]</td>
<td>- Needs radiation source [67] &lt;br&gt; - Strongly influenced by humidity; can lead to inaccuracies, requires air heating [67] &lt;br&gt; - Expensive [57] &lt;br&gt; - High power consumption [53], [54] &lt;br&gt; - Bulky [53], [54]</td>
</tr>
<tr>
<td>Optical Particle Counter (OPC)</td>
<td>Direct a laser beam at particles and count their number from the resulting light scattering [50], [69], [71]</td>
<td>- Widely used &lt;br&gt; - Can be small and inexpensive [17], [71] &lt;br&gt; - Less power consumption than methods above [17], [71]</td>
<td>- Needs steady airstream &lt;br&gt; - Often limited accuracy [58], [80] &lt;br&gt; - Can be expensive [57] &lt;br&gt; - Calibration needed for each type of analyte [50], [69]</td>
</tr>
<tr>
<td>Micro Electro Mechanical System (MEMS) particle Sensors</td>
<td>Particles land on a resonating material and the resonant frequency shift caused by this is measured [104]</td>
<td>- Small and inexpensive [12] &lt;br&gt; - Easy to manufacture [125] &lt;br&gt; - Low power consumption [4], [10]–[17]</td>
<td>- Require improved accuracy [4], [10]–[17] &lt;br&gt; - Lower sensitivity [4], [10]–[17]</td>
</tr>
</tbody>
</table>

Like optical devices, MEMS particle sensors require additional particle sampling and separation mechanisms, for example, a virtual impactor. It is important to ensure that deflected particles do not miss the sensing device, because of flow variations or temperature gradients [127]. Another drawback of many MEMS particle sensors, including FBARs is a reduction in mass sensitivity as particles keep settling on their surface. To extend the lifetime of these sensors a
mechanism for sensor regeneration should be found [106]. Section 2.3 will evaluate some commonly used particle sampling mechanisms and then move on to sensor cleaning.

2.2 Particle Selection and Cleaning Mechanisms for MEMS

2.2.1 Particle Separation and Deposition

Comparing MEMS sensors to FRM methods, one of their main issues is effective particle sampling [116]. For example, if a sensor has a theoretical detection limit of 100 ng of particles, at a particle concentration of 10 µg/m³ of uniformly distributed, uniformly sized particles in a cubic air chamber with a volume of 1 m³, this will require the sensor to successfully capture one in every 100 aerosolised particles. Considering a typical MEMS device is less than 1 cm long in each direction, which would also only be 1/10,000 of the bottom area and only 1/1,000,000 of the volume of said chamber, it can be easily seen that this is difficult to achieve with a sensor on its own. In addition, considering that many particles that would naturally settle on a MEMS sensor on an unfiltered air sample are going to be larger than ones of interest, the need for particle size separation is also obvious. Random particle settling may further lead to particle capture on different parts of the sensor in repeated measurements [128]. This is one explanation why the MEMS sensors discussed in section 2.2 and 2.3 have not yet been practically/commercially demonstrated to be capable of repeatedly discriminating levels of aerosolised PM at sufficiently low concentrations to be comparable to current air quality guidelines (around 10-50 µg/m³).

Even with significantly improved detection limits, MEMS sensors will thus require additional mechanisms to enhance measurement repeatability, particle collection and separation efficiency, and to not rely exclusively on natural settling of particles on the sensor surface [106], [108], [112], [129]. This somewhat complicates system design, but, with the current state of the art MEMS sensors, it is necessary to reduce detection limits for aerosolised particles of a certain size. In turn, the advantage is, especially for microbalances, an increased frequency shift at the same concentration of aerosolised particles, without needing to improve the microbalance itself [4].

To measure particle size distribution, particles can be analysed by microscope, which enables a detailed analysis. However, the time taken to analyse particle samples this way is a drawback [51]. Filters may also be used for reliable particle separation, as was highlighted in section 2.2.1, but this increases operation cost, because of filter saturation [4].
Diffusion Batteries are another way to separate particles [51]. They are created to find the diffusion coefficient of particles and can also be used to separate them according to their mobility [51]. However, this approach is mostly used for particles smaller than 0.1 µm where particle movement is dominated by diffusion [51].

Particles are commonly deposited or separated using mobility analysers [51]. Electrical Aerosol Analysers first charge the particles. Then they are passed through a chamber that only permits those particles with an electric mobility that does not exceed a set threshold to pass on to a concentration measurement chamber [59].

Differential mobility analysers are their successors that use bipolar diffusion charging [59]. Particle charge distribution is first brought to equilibrium by alpha-radiation and the particles are then charged by bipolar ions, also stemming from alpha radiation [130]. This is illustrated in Fig. 2.11.

Subsequently, remaining ions are removed, and the particles are separated based on their electrostatic properties and concentration can then be measured [130], for example by ramping up a voltage over time to measure response covering a range of particle diameters. This is done in Scanning Mobility Particle Sizers [59].

There are several issues with mobility analysers. Scan speeds are limited by the time taken after voltage application to the charged particles for a response to build up without distortion [131].

Fig. 2.11. Differential Mobility Analyser Schematic.
Furthermore, inaccuracies in charge distribution, for example because of non-spherical particles, multiple charging, and insufficient particle concentration for a charge distribution to establish can lead to significant measurement errors (with variations in measurements exceeding 20 percent) [131]. A device based on the diffusion charge principle to sense very small (<500 nm) particles was developed by Zhang et al. for a monodisperse aerosol [132] and then for a polydisperse aerosol [133]. It attempts to produce a compact, low-cost device, whilst proposing a real-time detection scheme with accuracy of about 12.2% compared to commercial electrostatic devices [133]. The device is however not designed for larger types of particulate matter.

“Corona-wire diffusion chargers” are used for unipolar particle charging in differential mobility spectrometers. An electrostatic shaft then captures the particles and measures the thus induced current to calculate particle size distribution [59]. Some low-cost particle sensors use this method [134]. However, particle charging can be inaccurate on these instruments [59]. Reasons include unintentionally charging some particles more than once [59] and differing particle attributes [4], [134]. According to Giechaskiel the fast integrated mobility spectrometer employs a neutralizer to charge the particles to “bipolar equilibrium” followed by parallel plates with an electrical field between them which separates the particles “into different trajectories based on their electrical mobility” [59]. A condenser then enlarges the particles and after illumination an image of the particle stream is captured for analysis [59]. Charge-based sampling mechanism often require high voltages [106], which is impractical on MEMS sensors. Achieving the correct voltage perturbations to actuate piezoelectric resonators is also difficult to combine with this type of particle deposition.

Some particle separation mechanisms rely on centrifugal forces and electrostatic forces jointly. Two examples are the Centrifugal Particle Mass Analyser and the Aerosol Particle Mass device [51], [59]. In these the centrifugal and electrostatic forces oppose each other and particles can enter the sensor element of the devices depending on their charge-to-mass ratio [59]. An advantage of these centrifugal distribution measurement techniques is that they do not accumulate particles over time. This reduces the need for device cleaning. However, these devices are not yet fully matured for commercial applications where particles are not size pre-selected, with issues remaining with regards to the monitoring of a variation of particle sizes [59].

Microfluidic channels, relying on size dependent particle aerodynamics have been researched for particle separation [106], [129], [135]. Such devices are called impactors [135].
They combine good separation capabilities with high potential for portable designs and as Li et al. demonstrated in 2018, they are generally consistent with design simulation, which is a helpful advantage for the development of this technology [136]. Impactors rely on particle inertia, which is strongly dependent on the particles’ aerodynamic diameter [137]. In these devices an airstream is directed to a nozzle, with an impaction plate located close to its outlet and two flow channels, often directed perpendicular to the inlet nozzle [135]. The impaction plate collects particles with high inertia (those with diameters higher than a certain cut-off diameter), while the remainder of the flow will continue along the perpendicular channels [137]. A virtual impactor works similarly but replaces the impaction plate with another channel [138], see Fig. 2.12. This is advantageous for very small devices, since virtual impactors do not require impaction plate coatings to avoid particle bounce and the resulting re-entrainment of filtered particles into the filtered airstream [135], [139]. Virtual impactors also do not require regeneration of the impaction plate.
A drawback of virtual impactors is their limited filtration accuracy. Some small particles will not follow the perpendicular channel, but rather the path taken by those particles above the cut-off diameter [138]. On the other hand, virtual impactors are simple and usually relatively cheap to implement [94]. They have been shown to be capable of separating particles with efficiencies more than 50% [51], [59], [94], [107], [129]. In a comparison by Giorio et al. impactors with collection filters for weighing are stated as a reliable a way to monitor PM regulations, while it was found that optical particle counters generally tend to overestimate or underestimate particle concentration, depending on whether ambient levels are generally high or low [140]. Jianwen Sun et al. developed a particle monitor consisting of an optical sensor and a microfabricated virtual impactor for PM2.5 and were able to successfully separate particles before the sensing stage of the monitor.
their setup [141]. Yuen et al. also tested an impactor in conjunction with an OPC and whilst the particle separation is shown to be effective, some issues with flow expansion between the impactor channel and the sensing chamber needed for optical sensors are pointed out, as some particles escape and are not detected because of this [142]. Impactors can be used in cascaded arrangements to allow for multiple stages of particle separation, which improves accuracy, and this has been tested as a deposition mechanism for resonant sensors repeatedly [140] [13] [143].

Mehdizadeh et al. used an impactor with a thermally actuated resonator in 2013, but operating frequency was limited to 2.87MHz [143] [144]. In 2017 Maldonado-Garcia et al. presented a MEMS resonator particle sensor manufactured on the front face of a silicon-on-insulator (SOI) wafer, with a virtual impactor manufactured on the same wafer by etching nozzles into the back of the device [145]. The impactor had a cut-off diameter less than 150 nm and was not suitable for PM2.5 monitoring. The device is shown to be capable of removing particles larger than 2 µm, but particle separation efficiency is not evaluated further [145]. The main upside of this design is its exceptional compactness and the requirement for only one wafer, while being able to combine multiple impactors and microbalances on a single chip [145]. This suggests excellent potential for mass-manufacture if the design is refined and characterised further. Djoumi et al. used a 150 MHz delay-line SAW device together with a cascade impactor and their setup successfully differentiated and detected PM10 and PM2.5 in real time [13]. According to Mehdizadeh et al. the impactor will also help the resonant sensors compensate for sensitivity variation depending on the particle location on the sensor surface [144]. The advantages of BAW devices in conjunction with impactors were pointed out by Villa-Lopéz, who designed a very compact PM2.5 monitoring device, consisting of a solidly mounted resonator and a virtual impactor, however this setup was not fully characterised [94]. Weng et al. also combined their TPoS MEMS particle sensor with an aerosol impactor in 2020, achieving a separation efficiency for PM2.5 of 51 % [107]. The disadvantage of virtual impactors is their requirement for precise control of airflow speed because their particle separation efficiency is heavily dependent on this [106]. In addition, higher air flow speeds also increase the power consumption of monitoring systems.

Wasisto et al. achieved sampling efficiencies of about 1.3 % in 2013 and 2015, by first applying an exclusively electrostatic sampling principle for naturally charged particles and then adding a virtual impactor, which was found to reduce sampling efficiency slightly (from 1.32 % to 1.25 %) [99], [100]. This is probably why Bertke et al. replaced the impactor with an integrated
microchannel for particle sampling in 2019 and 2021 [106]. They combined the mobility analyser sampling principle with a new MEMS cantilever sensor that has a channel etched directly out of the surrounding substrate [106]. Their new setup achieved a theoretical accuracy of about 14% and a theoretical sampling efficiency of 13 % [146]. The main drawbacks of this device are that it can only sample naturally charged particles, although this avoids multiple-charged particles, as well as the high absolute voltage required to attract particles of 20 V to 160 V on the 2021 version [106]. Bertke et al. also outlines the potential of applying electrostatic sampling to multiple sensors, which when applied at different levels can be used to separate particles. This reduces system size considerably, as it would otherwise require multiple impactor stages. An experimental analysis of this has not been carried out yet [106]. As Wasisto points out, the CANTOR was intended for use as a binary alert for high particle concentrations [100]. Whether the sampling mechanisms increase reading repeatability was not investigated in these works [99], [100], [106], [146].

Thermal precipitators use thermophoretic force for particle deposition [4], [147]. This force arises when a temperature gradient is created in a flow channel, which gives the particles of the gas on the warmer side of the particles a greater kinetic energy than on the colder side [147]. The dependence of the thermal velocity of the gas on the temperature gradient is indicated by (2.2), where $v_{th}$ is the thermal velocity, $k_B$ is the Boltzmann constant, $T$ is the temperature of the gas and $m$ is the molecular mass [148].

$$v_{th} = \sqrt{\frac{2k_B T}{m}} \quad (2.2)$$

The gas particles (in this case air) collide with the particles and push them towards the colder end of the temperature gradient. The path of the larger particles is altered more by this than that of smaller particles [149]. This way particles could be separated from the airstream passing the sensor [147]. Thermophoresis can thus be used to deposit particles on the sensing surface, see Fig. 2.13.
Fig. 2.13. Thermophoretic Particle Deposition Schematic.

Black et al. used a thermophoretic precipitator with an FBAR PM sensor and reported a PM2.5 detection limit as low as 18 µg/m³ [11]. The device weighs 148 g and is circa 245 ml in size with very low power consumption (under 100 mW) [11]. Sun et al. combined a thermophoretic device with a capacitive sensor and found it improved particle deposition by 84% [14]. External thermophoretic effects (natural temperature differentials around particles), one of the reasons for particle misplacement on microbalances, could also be eliminated this way [127]. Hao et al. developed such a thermophoretic precipitator for a surface acoustic wave resonator [16]. Liu et al. demonstrated it experimentally in a paper published in 2018, observing a frequency shift of about 4 kHz at 50 µg/m³ [120]. These works did not investigate repeatability of measurements.

Paprotny et al. combined a virtual impactor with a thermophoretic precipitator in an FBAR-based particle sensor reported in 2012 [150]. They demonstrated a response of their device down to a particle concentration of about 30 µg/m³ and estimated a theoretical detection limit for their device of 2 µg/m³ [150]. In a continuation of this work, Fahimi et al. miniaturised this design further by making a virtual impactor with vertically stacked, microfluidic channels in 2019 [129]. Their system consumes only 150 mW of power. With a footprint of 2.7 by 1.4 cm and a volume of only 760 nL, including drive circuitry (but excluding read-out capability), it is substantially smaller than other systems presented to date [129]. The device’s capability as a particle sensor is demonstrated at a particle concentration of 1.4 mg/m³, with the result indicating improved particle collection.
efficiency when the power supplied to the thermophoretic heater, i.e., its heat, is increased, although it is not clear from their results by how much the sampling setup improves the FBAR sensor and how well the sensor can discriminate varying particle masses with high repeatability [129].

2.2.2 Sensor Cleaning

In 2019, Singh et al. suggested using a virtual impactor for particle deposition and combining this with a hotplate above a MEMS resonator, to direct particles from the airstream onto the sensor and to potentially use thermophoresis for sensor cleaning as well [151].

Compressed air is a straightforward mechanism used, for example, to clean PM pollution off solar panels [152]. If applied at appropriate strength, it is also suitable for many MEMS particle sensors, although the provision of the compressed airstream for portable devices might be an obstacle. Wasisto et al. have demonstrated this using nitrogen gas to regenerate a cantilever particle sensor [99]. They also demonstrated the use of an ultrasonic cleaner with acetone or water and achieved cleaning efficiencies up to 99%, as well as that of a sacrificial layer, the replacement of which results in a regenerated sensor, albeit with low repeatability and high complexity [99], [106]. In 2021, Bertke et al. also achieved high efficiency cleaning using the ultrasonic method [106].

Microfan blowers used for air sampling could have a similar effect to the airstream cleaning and automate the cleaning process, but particle bonding of very small particles on the sensing area might be too strong for a sufficient cleaning effect with many of these fans [4], [99]. The OPC particle sensor SPS30 by Sensirion currently uses such a fan for particle sampling and sensor cleaning [82].

Solar panel cleaning with electrostatic forces was developed for space applications, but the potential to use the technology to clean very small particles of solar panels in large energy parks has also been pointed out [153], [154]. Choi et al., have applied this idea to a bridged paddle oscillator for particulate sensing [155]. They first use electrostatics to attract charged particles to the sensor surface and then after sensing they remove the potential attracting the particles, which are then repelled from the surface [155].

In 2017 Villa-Lopéz suggested applying a similar principle to solidly mounted resonator particle sensors, by designing a SMR with an integrated heater for device cleaning [94]. Yamamoto reported temperature requirements of 673 K to 923 K for successful thermal decomposition of PM, dependent on the size and material [156]. Lower temperatures could be feasible as well since
particulate filters in catalytic converters use thermal decomposition of particles for regeneration at around 573K for NO$_2$ [157].

Kim _et al._ demonstrated the use of vibration-based cleaning, as it is done in some cameras, on a lead zirconate titanate (PZT) bridged resonator in 2018 [158]. It required relatively high voltages of up to 18 V for sufficient acceleration for particle removal [158].

2.3 Chapter 2 Summary

Chapter 2.1 of this thesis has reiterated why it is necessary to reduce the exposure of humans to air pollution. It has long been established that better, cheaper, more mobile, and readily available particulate matter monitoring equipment is necessary to control the impact of air pollution on health and to meet air quality regulations. Section 2.1.1 to 2.1.4 have discussed commercial particulate matter monitoring devices, with emphasis on developments in the field of low-cost optical particle sensors. Particle sensors based on microelectromechanical systems (MEMS) are an area with substantial potential to resolve many of the issues currently facing better spatiotemporal resolution of pollution data. The current state of the art of MEMS particle sensors was discussed in section 2.1.5 and 2.1.6. Owing to their compatibility with low-cost CMOS integrated circuit manufacturing techniques, it has been concluded that bulk acoustic wave particle sensors based on piezoelectric thin-film technology warrant further investigation. Section 2.2 reviewed mechanisms for particle deposition and separation compatible with MEMS particle sensors to create complete, low-cost, reliable, PM measurement systems. The particle detection limit and reliability of bulk acoustic wave sensors were identified as the main limitation to their widespread application in low-cost PM monitoring. For this reason, this thesis will focus on improving the mass detection limit of bulk acoustic wave particulate matter sensors and methods to increase particle sampling efficiency. The CMOS compatibility of SMR-based devices will be explored by investigating the trade-off between standard fabrication process and quality factor of the devices. A novel solidly mounted resonator sensor manufactured in a complete CMOS-BAW process is presented in chapter 4 and evaluated in chapter 6. Temperature variations are the most relevant noise affecting the reliability of these devices. This work will attempt to use the temperature dependence of solidly mounted resonators to increase their sensitivity by integrating a microheater on-chip with the solidly mounted resonator. This device still relies on off-chip drive and readout circuitry, but further iterations may add this with relative simplicity. Methods to achieve reliable, repeatable real-time measurements with sensitivities to satisfy air quality regulations, thus creating a path towards successful
commercial exploitation will be of particular interest. In addition, the efficiency of thermophoretic sampling will be combined with novel SMR devices. Chapter 3 will continue to explain their principle of operation in more detail.
3 Solidly Mounted Resonator Sensors

Commonly used methods for particle monitoring were explained in chapter 2. Because the topic of this thesis is the use of solidly mounted resonator for particle sensing, this chapter discusses the working principle of acoustic wave devices for sensing applications. Furthermore, some important design considerations for their use in sensing systems are discussed in terms of sensor stability and readout mechanisms.

3.1 SMR Devices and Theory

3.1.1 Working Principle of Acoustic Resonator Sensors

The principle of operation of these devices is the piezoelectric effect exhibited in crystals like quartz or lithium niobate, as well as in thin films made of zinc oxide (ZnO) or Aluminium Nitride (AlN) for example [159], [160].

The piezoelectric effect is defined as “the induction of an electric charge in response to an applied mechanical strain” [161] or “the change of electric polarization proportional to the strain” [162]. Except for crystal class 29, the piezoelectric effect occurs within all crystal structure classes without a centre of symmetry [162]. Without a directly applied force, the centres of positive and negative charges inside the crystal coincide and cancel each other. When the crystal is deformed these centres move apart and a potential difference builds up between them, causing charges to appear at the material surface. This leads to the electric polarisation of the piezoelectric [163]. In a solid under deformation, atoms leave their original positions, and a restoring force arises, which attempts to restore the solid to its equilibrium state [164]. If this deformation varies with time so that each atom oscillates around its original position, it creates a propagating acoustic wave as a net effect [159], [164].

Hooke’s law is typically used to describe such elastic forces, where the mechanical stress $T$ is given by the applied strain $S$ multiplied by the elastic constant of the material $c$. The electric polarisation effect, in the case of piezoelectric materials, is considered by the electric field, $E$, multiplied by the piezoelectric constant of the material, $e$, as shown in (3.1) [164].

$$T = cS - eE$$

(3.1)

In single crystal piezoelectric these properties are typically anisotropic, in polycrystalline thin films it depends on the uniformity of crystal orientation [159]. It can also be seen from (3.1)
that deformation will change the value of $E$ and thus electric displacement, $D$, can be calculated from (3.2), where $\varepsilon$ is the permittivity of the material [159], [164].

$$D = eS + \varepsilon E$$

(3.2)

Thus, when alternating tensile and compressive forces are applied to opposing crystal faces, turning $S$ into a function of frequency, and they induce a standing wave, a sinusoidal voltage results at the crystals electrodes [159]. It is possible to reverse this effect by applying a voltage to the electrodes, to introduce strain to the crystal [162].

Following from this, piezoelectric crystals or films may be made to vibrate and hence propagate an acoustic wave by the application of a sinusoidal electrical signal [165]. A device that makes use of this is referred to as an acoustic wave resonator. The stress and the electric displacement calculated using (3.1) and (3.2) are affected further by elastic damping caused by temperature variation (thermoelastic damping), vibration frequency, vibration type and most importantly by acoustic loss into the surrounding air medium [164], which is exploited to turn the acoustic resonator into a sensor [94], [159]. This is achieved by placing the resonator in a circuit designed to generate such a sinusoidal signal. According to Sedra-Smith an oscillator circuit “consists of an amplifier and a frequency-selective network connected in a positive-feedback loop” [166]. An example of this, using the Butterworth-Van Dkye equivalent circuit configuration to model the resonator [118] [160], is shown in Fig. 3.1.
The resistance $R_m$ is used to model the mechanical losses in the device, $C_m$ and $L_m$ are used to model the motional inductance and capacitance of the mechanical resonance, while $C_0$ is used to model the capacitance of the resonator based on the capacitive characteristics of its structure and $R_0$ is used to model the acoustic losses into the resonator’s surroundings [94].

The series and parallel resonant frequencies (or the antiresonance and resonance) of the device are given by equations 3.3 and 3.4 [166]. In this thesis they are extracted directly as the
trough and peak in the impedance spectrum of the resonator devices computed across a broad frequency range. Generally, the parallel resonance point is preferred, since it presents an impedance peak, rather than a notch in the frequency spectrum, leading to better quality signals [167]. Specific circuit topologies are discussed in section 3.2.1 of this thesis.

\[
\omega_s = \frac{1}{\sqrt{L_mC_m}} \quad (3.3)
\]

\[
\omega_p = \frac{1}{\sqrt{L_m \left( \frac{C_mC_0}{C_m + C_0} \right)}} \quad (3.4)
\]

Here \(\omega_s\) is the series resonant frequency, \(\omega_p\) is the parallel resonant frequency, \(L_m\) is the inductance and \(C_m\) the capacitance of the mechanical arm of the oscillator and \(C_0\) is the capacitance of the parallel arm of the oscillator [93], [162].

The resonator quality factor, \(Q\), is a measure of the relative energy loss of a resonator per cycle of the acoustic wave. Increased mechanical damping from material deposition on the sensor surface such as the deposition of functionalised layers or particles reduce this value. It can be calculated from the resonant frequency, \(f_r\), divided by the resonator’s 3dB-bandwidth, \(\Delta f_{3dB}\), see (3.5) [52]. The equation may be adapted to use either the parallel or series resonant frequency and it can hence be derived from (3.3) and (3.4).

\[
Q = \frac{f_r}{\Delta f_{3dB}} \quad (3.5)
\]

Another method to extract the quality factor is the rate of change of phase method [168]. It uses the phase difference between the sinusoidal biasing signal’s application and the subsequent displacement of the piezoelectric [168]. The quality factor can be calculated based on the rate of change of phase method using (3.6) [168].

\[
Q = \frac{f_r}{2} \left| \frac{\delta \phi_r}{\delta f} \right|_{f=f_r} \quad (3.6)
\]

Here \(\delta \phi_r\) is the rate of change of phase of the phase lag at the resonant frequency, \(f_r\).

The Barkhausen criterion states that for the output signal of an oscillator to be sinusoidal, loop gain should be one and the gain’s phase should be zero [166]. Further, this criterion should
only be met by the circuit at one frequency to provide a simple sinusoid [166]. Any changes to the frequency setting because of changes in device’s material or geometric properties leads to a change in resonator equivalent circuit properties. This leads to a shift in output frequency, phase, or amplitude, because the Barkhausen criterion must always be upheld by the feedback circuit [165]. This property is exploited when employing acoustic resonators in sensing applications.

Acoustic waves generated in piezoelectric resonators can be divided in surface and bulk acoustic wave devices. Surface acoustic waves include Rayleigh waves, where particle displacement has both a normal and a parallel component to the substrate surface, as well as Love waves, with only a parallel component that is generated within a guiding layer on top of a piezoelectric substrate [159]. Rayleigh wave-based sensors are mainly used for gaseous applications whilst Love waves are generally used for liquid-phase sensing but suffer from high noise levels at high frequencies [169]. Another type of acoustic waves are Lamb waves created from interference of Rayleigh waves on the surface of a device and are a mixture of surface and substrate waves [165]. These waves form the basis of the sensing mechanism of surface acoustic wave (SAW) resonator sensors. A top-view of a two-port surface acoustic wave device is in Fig. 3.2 [170]. A pair of metal inter-digital transducers (IDTs) deposited on top of the piezoelectric substrate is used for excitation and detection of acoustic waves on its surface [171].

![SAW Resonator Schematic Top View](image)

**Fig. 3.2. SAW Resonator Schematic Top View.**

Thickness shear and longitudinal mode waves propagate through the substrate bulk as bulk acoustic waves [52], [165]. They form the basis of Bulk Acoustic Wave (BAW) Sensors like the Quartz Crystal Microbalance (QCM), which use a shear wave in a special cut of quartz substrate for mass sensing but are limited in their resonant frequency because of their higher minimum
thickness compared to sensors made from piezoelectric thin films [94], [115], [165]. Film Bulk Acoustic Resonators (FBARs) and Solidly Mounted Resonators (SMRs) are examples of thin film resonators [94]. FBARs consist of a thin film that are sandwiched between two electrodes, with air on either side, as depicted in Fig. 3.3. The low acoustic impedance of air assists in trapping the acoustic energy in the resonator and hence decreases losses [172]. SMRs use a stack of layers called an acoustic mirror or Bragg reflector underneath the thin film to confine acoustic energy [160], [172]. In an acoustic reflector the insulator and conductor materials are typically alternated, as they have low and high acoustic wave damping respectively [164]. The boundaries between the layers hence each cause partial reflection of the wave and aid the trapping of the acoustic energy in the sensor [94]. A cross section of a SMR is shown in Fig. 3.4.

![FBAR Schematic Cross-Section](image)

**Fig. 3.3. FBAR Schematic Cross-Section.**

![SMR Schematic Cross-Section](image)

**Fig. 3.4. SMR Schematic Cross-Section.**
SAWs are relatively cheaper to make than BAW sensors, since they require fewer steps to be manufactured, although the footprint of BAW sensors is smaller, requiring less space on a wafer [94]. The effect of either of these arguments on sensor cost depends on the manufacturing process and wafer material used.

Manufacturing constraints on the smallest achievable spacing of the inter digital transducers, \( s_{\text{IDT}} \) limit the wavelength of the acoustic wave \( \lambda_R \) for a given material specific speed of sound \( v_R \), as represented in (3.7) [159]. This limits the achievable resonant frequency of SAW devices.

\[
    f_0 = \frac{v_R}{\lambda_R} = \frac{v_R}{2s_{\text{IDT}}} \tag{3.7}
\]

Surface acoustic waves have very shallow surface penetration and hence SAW sensors are very sensitive to changes in surface properties [52]. The penetration depth, \( \delta \), of a SAW sensor is proportional to its acoustic wavelength. For the Rayleigh wave, which is the most widely used in sensing applications, it is approximately equivalent to the wavelength [159], [173]. Thus, the penetration depth may be estimated according to (3.8).

\[
    \delta = \frac{\lambda_R}{f_0} = \frac{v_R}{f_0} \tag{3.8}
\]

BAW sensors are limited in resonant frequency depending on the smallest achievable film thickness [94], [171]. The wavelength of the resonant frequency of a piezoelectric thin film is equal to twice its thickness. If \( v_R \) is the velocity at which the acoustic wave propagates through the film and \( d \) is its thickness, the BAW resonant frequency \( f_r \) is given by Equation (3.9) [111].

\[
    f_r = \frac{v_R}{\lambda_R} = \frac{v_R}{2d} \tag{3.9}
\]

The penetration depth of a FBAR sensor is given by (3.10). Here \( \rho \) is the density and \( \eta \) the viscosity of the medium on the resonator surface [174].

\[
    \delta = \sqrt{\frac{\eta}{\pi \rho f_r}} = \sqrt{\frac{\eta\lambda_R}{\pi \rho v_R}} \tag{3.10}
\]

Since the frequency is much larger than the other components, it can be seen from (3.8) and (3.10) that \( \delta \) is larger for the SAW device, because of the larger wavelength of the acoustic wave.
Thus, at the same resonant frequency SAWs are relatively more sensitive than BAW sensors at their surfaces [13].

Even though this is the case, BAW sensors can operate at higher resonant frequencies compared to SAW sensors in maximum achievable resonant frequency, which increases their absolute frequency sensitivity considerably [18], [52], [94].

In 1959 Sauerbrey came up with the following equation to express the shift in quartz crystal resonant frequency, $\Delta f_r$, when a deposited mass $\Delta m$ on the crystal surface changes its thickness, $d$, by $\Delta d$ [18]. This is shown in (3.11), where $\rho$ is the film’s density and $A$ is its surface area [111].

$$\frac{\Delta f_r}{f_r} = -\frac{\Delta d}{d} = -\frac{\rho A d}{\rho A d} = -\frac{\Delta m}{m} = -\frac{2f_r \Delta m}{\rho A v_p} = -\frac{2f_r \Delta m}{A \sqrt{\mu \rho}}$$  \hspace{1cm} (3.11)

The piezoelectric crystal or film’s shear modulus or elastic compliance, $\mu$, is the inverse of Young’s modulus, $Y$ given by (3.12) [162]. In anisotropic materials $Y$ is replaced by the elastic constant, $c$, in tensor form.

$$\mu = \frac{1}{Y} = \frac{1}{\rho v_R^2}$$  \hspace{1cm} (3.12)

As can be seen, frequency sensitivity, $\Delta f_r$, is proportional to the device’s resonant frequency, which in turn is inversely proportional to crystal thickness. Equation (3.11) can be modified into (3.13) by multiplying by 0.5 when the resonator is a thin film rather than a single crystal [175]. This was also used by Chu et al. in 2017 [127].

$$\frac{\Delta f_r}{f_r} = -\frac{\Delta d}{2d} = -\frac{\rho A d}{2 \rho A d} = -\frac{\Delta m}{2m} = -\frac{2f_r \Delta m}{2A \rho v_p} = -\frac{f_r \Delta m}{A \sqrt{\mu \rho}}$$  \hspace{1cm} (3.13)

It has been found that when thin films are sufficiently thin or sufficiently rigid modifications to (3.9) are not necessary [176]. It has been attempted to use devices made to resonate based on different transduction effects. For example, in 2009 Hajjam et al. used a thermo-electric transducer to ease manufacture compared to the integration of piezoelectric crystals or films, however they use a correction factor of $4\pi^{-1}$, which shows that piezoelectric resonators can be expected to have higher sensitivity at the same frequency [177].
BAW devices further have higher Quality factors and hence lower acoustic losses, which is especially important also at higher frequencies, than SAW devices [172]. Their power handling performance is higher, and they have superior temperature stability, as well as better electrostatic discharge [172].

Whether to use FBAR or SMR technology mainly depends on the design and manufacturing capability of the SMR’s acoustic mirror, since the Bragg reflector performance needs to be optimised for the desired resonant frequency [178]. The SMR’s acoustic mirror, however, is more robust than the FBAR membrane [178]. Since the topic of this thesis is the measurement of particulate matter, one of the issues with resonant sensors for particle sensing is particle removal. SMRs readily lend themselves for the addition of heaters for cleaning purposes [94] or the application of cleaning operations that might otherwise damage FBARs excessively.

AlN has become of interest for commercial BAW resonators, because of its better compatibility with standard manufacturing processes compared to ZnO or PZT [94][179][179][172]. One reason for this is the similarity of the coefficient of thermal expansion of AlN and silicon. AlN also has high thermal conductivity and electrical insulation [110]. Scandium Aluminium Nitride (ScAlN) is also being researched currently, because of its stronger piezoelectricity [180]. ScAlN can increase the electromechanical coupling of BAW sensors substantially. However, ScAlN yields lower $Q$ factors, because of the lower stiffness of ScAlN compared to AlN [180], [181].

Nevertheless, the roughness of the AlN can contribute to losses and reduce quality factor $Q$ [124]. Typically, vapour deposition or magnetron sputtering is used to manufacture polycrystalline aluminium nitride thin films [182]. Single crystal AlN deposition is currently limited by achievable film sizes. The electrodes should have high acoustic impedance to complement the Bragg reflector [182].

Spurious frequency modes can also be caused by manufacturing or design deficiencies of the resonator itself but can be avoided by careful electrode placement and appropriate design of drive circuitry. The electrodes are typically designed according to the apodisation (or windowing) method, to suppress spurious modes caused by reflections of transverse waves. The method is implemented by avoiding parallel edges of the top electrode. It increases $Q$ and electromechanical coupling [182].
It can also be seen from (3.10) and (3.11) that when used for particle detection, higher frequency devices are less sensitive to bigger particles than lower frequency devices. This is because larger particles also have lower resonant frequencies and thus larger penetration depth themselves. This was confirmed by Dybwad in 1985, who also pointed out the need for a coupling coefficient between particles deposited on the resonator surface and the resonator itself [109] and, for example, also further studied by Johannsmann [183], H. Zhang [184], Q. Zhang [184] and Villa-López [94].

3.1.2 Stability of Solidly Mounted Resonators

The Q factor of a solidly mounted resonator has been shown to decrease with increasing electrode size [185]. For a stable signal it is thus important to tune the dimensions of the BAW electrodes carefully. The resulting, low phase noise reduces the minimum detectable frequency shift of the sensor and hence its detection limit [52]. Nonetheless, if Q is made too high, it can lead to reduced sensor resolution [52]. It may thus sometimes be desirable to have larger sensor electrodes, which would also be beneficial for particle deposition, since, assuming the particles settle randomly on the sensor surface, a larger electrode increases the chance of particle capture.

Sauerbrey identified temperature dependence as the main limitation for quartz resonators [111]. FBARs and SMRs have also been shown to be very temperature and pressure sensitive. Zinc oxide and aluminium nitride FBARs and SMRs have been successfully used as temperature and pressure sensors [186] [187]. A main reason for the temperature dependence is the temperature coefficient of the piezoelectric film’s stiffness [18], [188]. This is because increasing temperature typically lowers the stiffness of the resonator and increases the damping experienced by the oscillating system, which lowers the resonant frequency of the device similarly to the effect of increased resonator size and mass [164]. FBARs are particularly sensitive to pressure variations, because of their stronger dependence on air instead of an acoustic reflector, both above the device and underneath the FBAR membrane [18], [186].

To reduce resonant frequency drift caused by self-heating effects and ambient temperature changes, as well as temperature gradients above the sensor caused by flow variations, thermophoretic precipitators, etc., direct resonator temperature control can be used [189]–[191]. This approach requires the addition of a heating element to the resonator and comes at the cost of increased power consumption [188]. It is commonly used in oven-controlled crystal oscillators; however, the heater must be added in such a way as to not isolate the sensor from its analyte. This
could even be exploited to change sensor selectivity without the need for physical alterations to the sensor [192]. Field effect sensors commonly do this for gas sensing [192], [193].

An approach by Pang et al. includes the addition of a capacitance-controlled cantilever, but this adds complexity to sensor design and manufacture [188]. A simpler method of compensation is the addition of additional temperature coefficient reducing layers [94], [188]. Temperature compensation can also be incorporated into the driving oscillator circuitry rather than into the resonator itself [194]. In 2012 García-Gancedo et al. designed a FBAR with resonant modes on the same resonator chip exhibiting opposing frequency behaviour to thermal noise and analyte [195]. An obstacle to this is the increased design effort required, as well as the need to track two modes.

Some designs use multiple resonator modes with different temperature dependences to discriminate between frequency shifts caused by particles and temperature. Both modes have the same frequency slope when a mass is deposited. When temperature changes on top of this, the slope of one mode will become steeper than that of the other, see for example a design by Mirea et al. from 2020 [191]. The approach does require the separate monitoring of two modes and thus enhances system complexity considerably.

BAW sensors are also highly dependent on humidity conditions [196], as this influences resonator rigidity [176], but condensation can also settle on the sensor surface.

A simple method to compensate for humidity, pressure and temperature changes is the use of a sensor specific compensation algorithm and an external reference sensor to monitor variations in these ambient conditions, as was done by Huang et al. [194]. Hu et al. propose a method considering the variability of resonator temperature dependence as the resonant frequency changes, which was found to effectively compensate for temperature variations [197].

Zhang et al. integrated a heater element under a zinc-oxide FBAR pressure sensor and were able to demonstrate a substantial reduction in temperature and humidity dependence of their FBAR [198]. They also increased the pressure sensitivity of their sensor by increasing its temperature [198].

Electromagnetic interference is another significant noise effect in piezoelectric resonators and can introduce undesired frequency components into the resonator output spectrum but can also act as additive noise at the desired resonant frequency, especially when this is close to commonly used communication bands [199]. Careful output filter design can help avoid this and especially
when the sensing parameter of interest is frequency, additive noise changing the resonant frequency amplitude should not be problematic if the correct resonant frequency is preserved. Placing the sensing device in a Faraday cage may help avoid electromagnetic interference, too.

Resonators are further sensitive to radiation and FBARs, for example, have been used as sensors for gamma radiation. Their resonant frequency is inversely proportional to radiation at, for example, about 9.3kHz/krad [200].

The photoelectric effect can induce currents when ultraviolet light is incident on the thin films, causing frequency to upshift [201]. This can be avoided by careful shielding of the BAW device from direct sunlight or other UV sources.

Common mode interference to sensors is commonly tackled by using two devices differentially, which will be discussed in more detail in section 3.2.2.

3.2 Acoustic Wave Resonator Circuitry

3.2.1 Drive Circuitry

Two common crystal oscillator configurations are the Colpitts oscillator and the Pierce oscillator circuit, which is shown under the Butterworth-Van Dyke model in Fig. 3.1 [166], [202]. They were probably developed from the Hartley oscillator but have shown better crystal oscillator performance and are thus preferred for FBAR oscillators [202]. Another benefit of these topologies is their high compatibility with modern CMOS designs [202]. They use a transistor to provide gain in a feedback loop for sustained oscillations and an RC network for biasing and frequency and amplitude control [166]. The Colpitts oscillator is an LC oscillator, however the inductor may be replaced with the resonant device to make a Pierce oscillator [94], [202]. This enhances its CMOS compatibility, although typically the resonator is placed off-chip [4], [129], [150]. Devices combining acoustic wave resonators and oscillator drive circuitry are in development, with completely integrated processes becoming more widely available (for example SilTerra’s CMOS technology) [4], [96], [97].

Vittoz et al. designed a Pierce oscillator including output amplitude and voltage regulation through additional circuit elements [203]. Vittoz et al. also point out the need for compensation for the voltage dependent Miller capacitance in the gain transistor, the lower influence of which is one of the advantages of Pierce type Colpitts based oscillators, because of the grounded source or grounded emitter reducing bulk modulation effects, even though it does require two pins for the
resonator rather than one [203]. The Miller Capacitance introduces frequency instability by affecting overall circuit capacitance and the matching of the resonator resistance to the negative resistance in the oscillator circuit required, since the real and imaginary parts of the circuit transfer function must be zero for oscillations to start [203]. For example, a 0.5 mW Pierce oscillator was designed for 3GHz by Pillai et al., but oscillations were not observed in this, caused by a lack of compensation for the Miller capacitance effects [204].

Nomura et al. attempted to solve this by introducing an emitter follower or a common base circuit, placing another voltage dependent Miller capacitance near the gain transistor’s one, leading to a lower equivalent Miller Capacitance affecting the negative oscillator resistance and the circuit’s frequency and enabling the design of a negative compensation resistance. This effectively improves the high frequency performance of the Colpitts Oscillator [205]. Power consumption is a drawback with Colpitts type oscillators [203]. Abdolvand et al. suggest a circuit reported to use only 350µW of power, but this was not tested in the UHF range. Pierce oscillators are known to provide good high frequency performance [203].

Azadmehr et al. also found common gate Colpitts oscillators to be better at preserving oscillator signal strength during mass loading, although this reduced the frequency shift from mass loading as well [206]. If used for particle sensing it may also not be desirable to reduce signal damping from particles. On top of this their mass would likely have to be very large to achieve complete signal cancellation. BAW sensors for liquid-phase environments may require a consideration of this, because of stronger signal attenuation into the liquid [191], but in this work this does not apply. The main reason common emitter circuits are typically used in radio frequency crystal oscillators is their low phase noise and resulting high Q and superior ability to resonate stray resonances. In a common emitter amplifier circuit, the feedback and thus input capacitance is large. This leads to a smaller bandwidth of the oscillator and thus lower phase, noise, increasing Q [166], [207].

Nelson et al. developed a custom, integrated FBAR oscillator consuming 50µW, using a high resistance FBAR to reduce current consumption and then weakly forward biasing the substrate bulk to achieve lower operating voltages than would otherwise be possible [208]. This design does come at the cost of significantly increased design and manufacturing complexity compared to standard Pierce oscillators. Design complexity of the FBAR is also increased by the maximisation of the FBAR’s parallel resistance [208].
A Pierce oscillator designed by Peng et al. uses capacitor arrays to allow for oscillator frequency adjustment, however the design is for 16 MHz and the suitability of capacitor banks for UHF oscillator tuning, where capacitance accuracy and stability is crucial, is not assessed [209].

Thomas and Villa-Lopéz successfully used Colpitts and Pierce Oscillators for 870 MHz and 1.5 GHz SMRs respectively for particulate matter and volatile organic compound sensing [12], [94], [210]. Their circuit uses the common emitter topology.

Since most resonant particle sensors focus only on the frequency shift to sense particles, beyond considerations regarding circuit integration, oscillator circuit topologies and especially the optimisation of their performance are often not investigated with the utmost importance [206], [211]. In fact, none of the designs mentioned in this section discuss this topic. This is most likely because currently the focus of the field of resonant sensors is on reducing the detection limit of the sensors itself.

The literature discussed in section 2.1.6 often does not include discrete drive circuitry and relies on specialist equipment like spectrum or vector network analysers (VNAs) instead. The next section (3.2.2) discusses some more practical alternatives to this read-out approach. Since the specific readout mechanism is not focus of this thesis it will focus only on topologies used for piezoelectric acoustic wave sensors.

3.2.2 Mixer Readout Circuitry
Spectrum analysers require a working drive circuit for the FBAR to be capable of measuring a signal. VNAs are capable of exciting and reading the frequency response of resonant devices simultaneously and at high speed across a broad frequency spectrum (impedance-based measurement) [212]. Both methods enable detection of signal loss as well as frequency shift. Especially with the ultra-high frequencies required for FBAR particle sensors to achieve the necessary sensitivity, these devices for direct very-high and ultra-high frequency measurement (VHF and UHF) are so expensive, alternatives must be found to make a viable low-cost system [4], [18], [125], [212]. For example, in the MEMS PM2.5 monitor design presented by Fahimi et al. in 2019, a FBAR particle sensor is driven by a CMOS Pierce oscillator. The schematic design of this chip was presented in 2013 by Paprotny et al. [150]. As mentioned in section 2.3 this setup is exceptionally compact, combining sampling and sensing elements in a package smaller than 760 nL. However, it uses a Keysight N9342C for Pierce oscillator frequency measurement (the authors state they are working on a new data recording mechanism) [129]. Two cheaper, more portable
examples of a spectrum analyser and a VNA are the N9342C’s successor, the Keithley N9912A (400 mL volume, 2.8 kg, 12W, battery powered, on sale for about $7300 [213]), and the picoVNA 106 (300 mL volume, but requires separate PC and screen, 1.9 kg, 22 W, mains powered, on sale for about $6500 [214], [215]) respectively. The most popular way to enable low-cost FBAR readout has been to simply find ways to translate the FBAR response to a signal at a lower frequency, while maintaining an effect of particle deposition on this signal.

Perhaps the most straightforward way of reducing oscillator frequency is to use a frequency divider chip. This was done, for example by Jin et al., who lowered a frequency of about 1.9 GHz down to 7.5 MHz by dividing it by 255 [216]. The simplicity of this topology is its obvious advantage, the reduction in frequency shift by the division factor is its main disadvantage. If there is noise present at the lower frequency level, the shift caused by particle deposition becomes more difficult to distinguish. The circuit is also more vulnerable to thermal noise than the topologies discussed below.

It has been demonstrated by several works, that sensor readout can be achieved by using the oscillator with a stable frequency reference and a mixer circuit. For example, Thomas et al. did this in 2016, as did Liu et al. in 2019 [12]. In these works, the mixer performs a convolution operation on the signal from the generator of the reference frequency and the signal from the oscillator with the reference circuit [4], [18], [211]. The output of the mixer is a signal containing the difference and the sum of the two signals. A low-pass filter can be used to remove the sum, resulting in a much lower frequency signal, depending on the level of the reference frequency [211]. The advantage of this configuration is that it fully preserves the absolute change in frequency of the sensing oscillator, while the much lower frequency signal is simpler to measure with a frequency counter [4], [18], [211]. This is a way of measuring the sensor’s frequency shift caused by the settling of particles [4], [12], [16]. Frequency measurement be done cheaply with a timer and a counter circuit on a microcontroller of sufficient speed (e.g. Teensy) or FPGA boards [4], [12], [18], [212], [217]. A schematic of this is shown in Fig. 3.5 [18].
A comparator or level shifter may be required for the oscillator voltage to reach the logic level of the microcontroller [211]. The reference can be a frequency generator, such as a voltage-controlled oscillator (VCO; at UHF available for around $20) or a second oscillator circuit with another FBAR. The latter approach is also commonly adopted to reduce common mode noise in the readout signal [4], [12], [70], [94], [120], [218]. For example, thermal, air flow or humidity noise that causes frequency drift on both devices can be tackled this way. The main challenge in this case is creating comparable ambient conditions without exposing both sensors to particles [4], [12], [16], [117]. Thomas et al. attempted to do this by adding a very thin, thermally conducting cap with inlets at the sides, to allow for humidity exposure [12]. This will however not work when, for example, there is a flow over the sensing resonator that the cap blocks. Black et al. (also White et al.) tried an active and a passive mixer differential setup in 2008 but found both not to be fully capable of cancelling common mode noise and they thus ended up not using a reference device for sensor read-out [11], [218]. Manufacturing variations that lead to different frequency coefficients of temperature, etc. are the main reason for this [218].

Thomas also designed a single-chip implementation of oscillator circuits for sensing and reference, mixer, low-pass filter, and comparator [210]. However, this device was shown to saturate outside a certain frequency range [94]. This highlights the challenges of designing such a system at UHF. There is a trade of between the desire to have low power consumption and the resonator Signal-to-Noise ratio. Another limitation is the clock speed and size of the microcontroller timer circuit. The clock speed needs to be large enough to reliably sample the comparator output, while the timer circuit must be kept from overflowing during the possible measurement intervals.

One way to reduce variability between sensing and reference signal is to use the resonant modes of a single chip as a self-reference [113]. Waj et al. designed a SMR with different
sensitivity to particle deposition for each mode in 2019 [113]. The oscillator circuits need to be
designed to support this.

Another readout circuitry that has been proposed for normal resonator sensors, but also
specifically for dual mode resonators is the phase-locked loop (PLL) [212], [219], [220]. These are
commonly used in communications applications. A phase detector circuit outputs a voltage signal
proportional to the frequency difference and this can be easily measured with a standard analogue-
to-digital converter. For this it is more important than for the mixer topology that the circuit has
low phase noise. PLL readout setups are considerably more complex than the mixer topology, but
their output signal can potentially be sampled faster than many frequency counters operate [212].
They require additional components; the use of a VCO is typically required, because of the noise
present in an exposed FBAR reference and they need to be very carefully calibrated [167], [212],
[219]. Both frequencies need to match initially. The voltage slope from the phase detector that
controls the VCO needs to be matched to the FBAR response to particle deposition, too. Liu et al.
have demonstrated such a design in [167].

Mixer circuits can be driven differentially and single-ended, the differential implementation
benefitting from superior common-mode noise immunity, whilst being more complex to design.
Voltti et al. compared passive and active mixers and while passive mixers are simpler in operation,
active mixers preserve the signal-to-noise ratio in a superior manner and have positive rather than
negative conversion gain [221]. Since resonant sensors are low-power and very noise sensitive, a
balanced, active mixer configuration recommends itself, but the added value of this should be
carefully considered when signal strength preservation is not of paramount importance [206]. This
may well be the case in a system where only a simple measurement of frequency difference between
two circuits is needed. Discrete, differential PM monitoring systems have typically used passive
mixers [4], [14], [33], [88], [217]. In addition, some mixers, but also level shifters or comparators
used to make the frequency difference measurable with standard microcontrollers remove any
FBAR impedance information from the signal strength of their output anyway.

If desired, cheap signal amplitude detection can be achieved with techniques borrowed from
communications engineering, such as the demodulation of amplitude modulated signals [211]. A
FBAR oscillator circuit, if sufficiently sensitive, experiences amplitude modulation after particle
deposition and this can be measured with cheap envelope or product detector chips.
3.3 Chapter 3 Summary

This chapter has briefly outlined the working principle of acoustic wave sensors. The compatibility with standard manufacturing processes of solidly mounted resonators specifically was outlined in chapter 2, but particle sensors making full use of this have not yet been evaluated extensively. Chapter 3 discussed some common approaches for sensor drive and frequency measurement. Because the work in this thesis focuses on SMR frequency shifts caused by particle deposition it was found that drive and readout circuitry do not need to be fully optimised and integrated at this stage. For this reason, the work will focus on differential frequency readout using a passive mixer circuit and only consider shifts in signal amplitude briefly in chapters 4 and 6. The functioning oscillator drive and readout circuitry designed by Thomas et al. [12], was adapted for the novel CMOS SMR with only minor adjustments made. Chapter 5 will explain the setup in more detail.
4 Design and Fabrication of a Thermally Modulated CMOS SMR

In chapters 2 and 3 bulk acoustic wave (BAW) sensors based on complementary metal oxide semiconductor (CMOS) technology have been introduced as a promising option for the realisation of low-cost particulate matter sensors. Temperature dependence of solidly mounted resonators (SMRs) has been identified as an undesirable effect that must be controlled, but once this is achieved the devices can be exploited for sensing applications. To investigate this beyond theoretical assumptions, the physical realisation of a low-cost (mass-manufacturable), integrated resonant microsensor with temperature control was explored.

Chapter 4.1 first explains the design of a novel CMOS SMR with an integrated microheater, based on the fully integrated CMOS-BAW process by SilTerra Malaysia [125]. It then continues to describe proof-of-concept simulations used to verify the design prior to device manufacture for both general resonator functionality, as well as particle sensing with the novel SMRs. The sensor was then fabricated by SilTerra Malaysia and the physical device, as well as its general characterisation are presented in section 4.2.

4.1 Design and Simulation of CMOS SMR

As it was introduced in section 3.1, a BAW device is comprised of a piezoelectric thin-film with metal electrodes on either side. In the case of SMRs, the device has the addition of an acoustic (also Bragg) reflector stack underneath the bottom electrode to mimic the excellent acoustic reflection properties of an air boundary while giving the device greater ruggedness and simpler manufacturing than a membrane-based design would offer. This is an important advantage for real-world deployment of the device as a particle sensor.

The Bragg reflector works by creating boundaries between layers of low and high acoustic impedance that if designed correctly, result in repeated constructive interference upon reflection of the remaining acoustic wave at each boundary. Because the wave is a standing wave and with high reflection at the boundary between the resonator and the surrounding air at the top electrode, more of the acoustic energy is trapped inside the resonator this way, compared to mounting the device on a bare substrate, which also leads to an increased Q factor. The advantages and disadvantages of this are discussed in chapter 3. An obstacle to SMR integration with standard CMOS processes, especially if the BAW device is intended for communication purposes rather than sensing, is the design of the Bragg reflector layer stack since optimisation requires precise control of layer thicknesses and materials.
4.1.1 SMR Design

CMOS processes commonly use oxides of silicon, for example silicon dioxide (SiO$_2$) or fluorosilicate glass, for their insulating dielectric layers (simply referred to as oxide henceforth). These oxides are also very suitable as low acoustic impedance layers in acoustic Bragg reflectors [94]. This is exploited in the fabrication of the SMR devices using the 180 nm CMOS process as described in this chapter.

To reduce the number of post processing steps required for the realisation of CMOS compatible BAW devices, it is desirable to utilise as many layers as possible from the CMOS stack for the SMR design. Because of this, the metallisation layers, used for wire interconnects in CMOS circuits, are going to serve as the high acoustic impedance material of the Bragg reflector. In addition, to implement temperature control and explore temperature nodulation, the integrated microheater will also be implemented in a CMOS metallisation layers between the Bragg reflector and the CMOS substrate [125].

To optimise the performance of the reflector, the ratio of the high and the low acoustic impedance should be maximised. Equation (4.1) can be used to calculate the acoustic impedance, $Z_L$, of the material layer based on the material density, $\rho$, and the speed of sound in the material, $v$ [94].

$$Z_L = \rho v \quad (4.1)$$

Bragg reflectors typically use high density metals or insulators like aluminium nitride. Even though a fully dielectric design can reduce parasitic effects [52], metal layers generally allow higher heat conduction which can be advantageous however, typical CMOS processes do not offer fully dielectric layer stacks [94], [125], [223]. Thus Table 4.1 only lists some commonly used Bragg reflector, as well as CMOS metallisation materials and their properties for general comparison [94], [223].
Table 4.1. Material Properties of Different Metals.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (kg/m³)</th>
<th>Speed of Sound (m/s)</th>
<th>Resistivity (Ωm)</th>
<th>Thermal Conductivity (W/(mK))</th>
<th>Acoustic Impedance (Pa s/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tungsten</td>
<td>19300</td>
<td>5200</td>
<td>52.8n</td>
<td>173</td>
<td>100</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>10200</td>
<td>6340</td>
<td>53.4n</td>
<td>138</td>
<td>65</td>
</tr>
<tr>
<td>Copper</td>
<td>8960</td>
<td>3810</td>
<td>16.8n</td>
<td>401</td>
<td>34</td>
</tr>
<tr>
<td>Gold</td>
<td>19300</td>
<td>2030</td>
<td>22.1n</td>
<td>318</td>
<td>40</td>
</tr>
<tr>
<td>Aluminium</td>
<td>2700</td>
<td>6500</td>
<td>26.5n</td>
<td>237</td>
<td>18</td>
</tr>
<tr>
<td>Silicon Dioxide</td>
<td>2200</td>
<td>5710</td>
<td>10¹⁵</td>
<td>1.3</td>
<td>13</td>
</tr>
</tbody>
</table>

Commonly used materials for SMR Bragg reflectors are molybdenum and tungsten [52]. These would also be the most suitable materials for the integrated microheater from Table 4.1, because of their higher resistivity that would lead to higher conversion of electric energy to heat. It is clear from this that tungsten, for example, would be a better choice as a high acoustic impedance material for the metallisation that would also be a better solution for the microheater. This also means an increased loss of electrical power and makes these materials less desirable for wiring in CMOS processes. Because of their high electrical and thermal conductivities, the most used CMOS metallisation materials are alloys of aluminium or copper.

As was stated above, the design requirement for this research mainly focuses on the ability to detect particles via SMR frequency readout on a low-cost CMOS compatible sensor. The resonant frequency was also desired to be between 1.5 and 2.5 GHz, to achieve good sensitivity and to function with existing oscillator circuitry. Power consumption and SMR quality factor were trade-off considerations if the simulation indicated the designed SMRs could be used as particle sensors. The SilTerra CMOS-BAW 180 nm process was identified for the design of a custom SMR with integrated microheater, offering aluminium metallisation by default. This in turn meant that instead of using the recommended Bragg reflector stack layer thickness of a quarter of the acoustic wavelength [52], [94], the stack layers were defined at thicknesses of 530 nm for the aluminium and 850 nm for the oxide, as available with the standard CMOS process. This implied a trade-off between the low-cost fabrication and ideal Bragg reflector design. The same was true for the metal electrodes, which had to be designed in aluminium, rather than high acoustic impedance...
molybdenum, with thicknesses of 400 nm and 350 nm [125]. Following these considerations, the following layer stack was designed, as shown in Fig. 4.1.

![Layer Stack Diagram]

**Fig. 4.1. CMOS-BAW SMR Cross-Section** [18].

To ensure further design efforts were not wasted, a short feasibility study for the CMOS-BAW was carried out using a simple, one-dimensional SMR model was assembled in MATLAB 2019b testing a variety material parameters and layer thicknesses over a wider frequency range with low computational effort. The model was based on the Mason model, similar to that employed by Mirea or Villa-Lopéz in 2017 [52], [94]. A schematic is shown in Fig. 4.2.

![Mason SMR Model Diagram]

**Fig. 4.2. Mason SMR Model.**
It modelled the piezoelectric layer as a parallel plate capacitor with capacitance \( C_0 \) based on (4.2).

\[
C_0 = \frac{\varepsilon A}{d_p} \quad (4.2)
\]

The piezoelectric properties of the resonator were included based on conversion from a transformer with \( h \) coils (4.3), where \( e \) is the piezoelectric constant and \( \varepsilon \) is the permittivity of the material (see section 3.1.1).

\[
h = \frac{e}{\varepsilon} \quad (4.3)
\]

The Mason model uses an electric equivalent circuit analogy (voltage \( V_a \) equals mechanical force, \( F \) and current \( I_a \) equals acoustic velocity, \( v \)) to model the propagation of the acoustic wave through a material [52]. Matrix notation may be used for this, see (4.4) [52], [94].

\[
\begin{pmatrix}
F_o \\
v_o
\end{pmatrix} = \begin{pmatrix}
1 + \frac{Z_T}{Z_S} & 2\frac{Z_T}{Z_S} \\
\frac{1}{Z_S} & s + \frac{Z_T}{Z_S}
\end{pmatrix} \begin{pmatrix}
F_i \\
v_i
\end{pmatrix} \quad (4.4)
\]

\[
Z_T = jZ_L \tan \left( \frac{\pi f d}{v} \right) \quad (4.5)
\]

\[
Z_S = -jZ_L \frac{\sin \left( \frac{2\pi f d}{v} \right)}{} \quad (4.6)
\]

The Bragg reflector was modelled as a cascaded stack of the 4 by 4 matrix in (4.4), defined by (4.5) and (4.6) for each layer. In these \( Z_T \) and \( Z_L \) express the mechanical impedance, \( f \) is the frequency and \( d \) is the layer thickness [52], [94]. The model assumed dry air at the top electrode. The piezoelectric layer (AlN) was assumed to be 2 \( \mu \)m thick. The electrode size was set to 350 \( \mu \)m \( \times \) 350 \( \mu \)m. The temperature was fixed at room temperature. The result of this simulation is shown in Fig. 4.3.
The model suggests the sensor would have a prominent resonant frequency peak at approximately 2.1 GHz, within the desired range. The design thus warranted further investigation, to get a better understanding of resonator performance characteristics than a one-dimensional model can provide.

To do this, the MEMS module of COMSOL Multiphysics 5.4® was used to create a two-dimensional finite element model of the solidly mounted resonator layer stack in Fig. 4.1. At this point the thickness of the AlN layer was changed to 1.3 µm thickness to better fit the BAW process. The reflector stack was built with three metal layers as before, see Fig. 4.1. The 2D model is shown with a cropped substrate in Fig. 4.4.

![Mason Model Output](image.png)
In the frequency domain it simulated the vertical cross-section under the top electrode of the BAW resonator including the Bragg reflector and 400 µm thick substrate under 1 W perturbations across the electrodes. The model uses (3.1) and (3.2) to calculate the resonator’s admittance across the defined frequency sweep (for the result shown in Fig.4.5 this is 2000 to 2010 MHz at 100 kHz resolution). The device’s Bragg reflector was likely to allow some leakage of acoustic energy into the substrate. Since the sensor was primarily tested when mounted onto a FR4 PCB, simulations were done with and without simulating a 1.6 mm thick board underneath the substrate. Based on this it was assessed whether additional mitigation mechanisms, such as mounting the sensor onto a PCB hole, might be necessary. Because the main limitation to previous BAW particle sensors was their sensitivity, it was decided to use a larger sized active area for this SMR (350 µm × 350 µm) compared to other recent attempts to make SMR particle sensors, for example, the 200 µm × 200 µm used by Thomas et al. [12]. The aim of this was to increase particle sampling efficiency and sensitivity to surface conditions, as was explained in section 3.1.2.
According to the result in Fig. 4.5, the resonant frequency of the SMR with 1.3 µm AlN is approximately 2,009 MHz. The two-dimensional study predicts a value for the S21 scattering parameter of the sensor of around -8 dB. S21 is reduced by 0.5 dB to -7.5 dB and the resonant frequency shifted down to approximately 2,004 MHz after the addition of an FR4 PCB. This suggests that there is some leakage of acoustic energy through the substrate into the PCB. However, since the device only seems to perform slightly better and at a resonant frequency that is only 0.25 % lower when it is mounted straight onto a PCB, it was deemed that the sensor will operate satisfactory and can be bonded to the Pierce oscillator PCB.

Based on these modelling results it was judged that SilTerra’s CMOS-BAW technology could be used to fabricate a resonator that can be exploited for particulate matter measurements. However, the thermal behaviour of the sensor still had to be evaluated before green-lighting sensor fabrication. The following sections will focus on the three-dimensional simulations of the chip.
4.1.2 3D Thermal Simulation of SMR

The proposed design of the SMR includes the integrated microheater in the bottom metal layer as shown in Fig. 4.1. As was explained in section 4.1.1, the devices will be fabricated in a standard CMOS process, thus the material for the heater had to be aluminium, which is not ideal. Additionally, microheaters are usually manufactured on thin membranes or even fully suspended in the air, to reduce conductive heat loss, especially into the substrate. This would require back etching of the resonator for this process, which was not available during this manufacturing run since the risk of device breakage would have been too high. It was, however, possible to back grind the substrate to reduce its thickness from 720 µm down to 400 µm. Regardless of this, the heater needed to be capable of uniform heat distribution across the sensing area and particle deposition should have a noticeable impact on the temperature of the active area of the piezoelectric layer.

The device was also required to operate powered by a Teensy 3.6 up to around 353 K for temperature modulation of the sensor, to avoid evaporating volatile particulates during testing [125]. It was also desired that the sensor should be able to exceed 573 K for potential extensions of the temperature range used in future work [125], [224]. Because of this it was important to design a device with sufficient power handling capability and thus with trackwidths that do not burn out too easily. The latter is also important to improve the uniformity of heater temperature, counter electromigration [225] and increase the longevity of the device, especially should its temperature be modulated during operation. One benefit of aluminium is that its low resistivity leads to insignificant loss of power along the tracks needed to connect the heater to the pads for wire-bonding.

Different heater designs were considered, one of the most used is that of the simple meander heater. The meander design suits this work mainly because it has been shown that it can achieve similar temperatures and heating speeds to other designs but at lower power consumption and resistivity [226]–[228]. Resulting temperature uniformity is typically lower, but the conductive heat loss through the reflector stack should make this effect less important. An inherent advantage of the designed structure is that the high thermal conductivity of the aluminium Bragg reflector layers should improve the uniformity of the temperature of the active area. From a thermal performance perspective, it thus serves a similar purpose to metal heat spreading plates that have been used in past designs, for example those by Iwaki [225]. Typical width of the meander tracks range up to a few microns for temperatures comparable to those desired in this application [225]–[228]. The layer stack including the microheater will have to be capable of handling higher power
input compared to membrane devices because of conduction losses into the substrate. To be on the safe side the heater tracks were thus set to 10 µm width, with the heater length set to the length of the active area of 350 µm [125]. Track thickness was set by the process to 530 nm. The designed resistance of the microheater was thus approximately 52 Ω. 5 µm tracks were also designed for comparison. A schematic drawing of the meander heater design is shown in Fig. 4.6.

![Designed Meander Heater](image)

**Fig. 4.6. Designed Meander Heater.**

As with the SMR structure, the behaviour of the heater was also simulated prior to mask manufacture. A one-dimensional model was deemed too inaccurate for this and, while the two-dimensional model allows the simple addition of out-of-plane heat loss, simulating the heater structure accurately is difficult without knowing exactly how much electricity the design converts to heat beforehand. Because of this, a three-dimensional model of the resonator was also created using the Joule Heating interface of COMSOL Multiphysics 5.4®. Interfaces from the MEMS
module and the Heat Transfer module were combined to model the device under external natural convection. A screenshot of the meshed three-dimensional model with an area of 1 mm by 2.5 mm and a substrate thickness of 400 µm is shown in Fig. 4.7.

Fig. 4.7. Three-dimensional COMSOL Model of SMR.

The Joule Heating interface of COMSOL was used to model the electromagnetic heating of the microheater, as it has shown to yield accurate results [225], [229]. The FEM model assumed three heat loss mechanisms affect the sensor temperature: conduction, $P_{\text{conduction}}$, convection, $P_{\text{convection}}$, and radiation, $P_{\text{radiation}}$. The standard equations used for these are given below by (4.7), (4.8) and (4.9) [230].

$$P_{\text{conduction}} = -k_{\text{thermal}} T$$  \hspace{1cm} (4.7)

$$P_{\text{convection}} = h_{\text{conv}} (T_{\text{ambient}} - T_{\text{surface}})$$  \hspace{1cm} (4.8)

$$P_{\text{radiation}} = \varepsilon_{\text{emissivity}} k_B (T_{\text{ambient}}^4 - T_{\text{surface}}^4)$$  \hspace{1cm} (4.9)

Here $k_{\text{thermal}}$ is the thermal conductivity of the material, $T$ is the local temperature at the relevant point in the FEM model, $h_{\text{conv}}$ is the convective heat transfer coefficient, $\varepsilon_{\text{emissivity}}$ is the surface emissivity of the material and $k_B$ is the Boltzmann constant, $T_{\text{ambient}}$ is the ambient
temperature (293 K) and $T_{\text{surface}}$ is the surface temperature of the device at the relevant point in the FEM model [230]. The Heat Transfer module of COMSOL was used to calculate $h_{\text{conv}}$ for external natural convection into air at 285 K at the upside and downside of horizontal plates, as well as for vertical walls using the height and characteristic length (area/perimeter) of the SMR. It uses the same equations that are described in Çengel’s book from 2006 [230]. The sensor was assumed to be a perfect black body. The true surface emissivity of the oxide is probably closer to 0.7, but this was rounded up to 1 since radiation losses are typically relatively low compared to the other sources of heat loss [225], [229]. The temperature coefficient of resistance (TCR) of the microheater material was also included. A steady-state study was used to calculate that a current of 60 mA (approximately 4.48 V) was sufficient to achieve almost 413 K resonator temperature. Figure 4.8 shows the temperature of the sensor surface.

![Temperature distribution in COMSOL SMR](image)

**Fig. 4.8. COMSOL SMR with 60 mA Heater Current.**

The desired operating temperature of the SMR should thus be achievable well within the 850 mW power supply limit of the Teensy 3.6. Additionally, the device heats up very uniformly, the average temperature of the active area and its maximum temperature were calculated to be within 2 K or approximately 1 % of each other. Unlike membrane heaters, the whole substrate can be seen to heat up in Fig. 4.8. According to the COMSOL simulation the heater consumes 268.8
mW total power at 60 mA current. The main heat loss mechanism is conduction, as expected, with 231.2 mW heat loss. The remaining power loss is divided into 37.1 mW convective heat loss and only 0.6 mW heat loss from surface radiation.

For the first round of planned practical experiments, it was decided to test the sensor at an elevated temperature not too far above the ambient. This is to ensure several repeat particle sensing tests could be carried out without device deformation and to avoid breakage. Heating the sensor to 318 K, approximately twice the ambient temperature in the test chamber (around 395 K), should be sufficient to show a proof-of-concept for a desired sensitivity increase through temperature modulation. The sensor will be tested practically with Arizona test dust of type ISO 12103-1 A1. Simulations in COMSOL were carried out to estimate whether particle deposition would alter the thermal behaviour of the sensor. A solid layer of quartz (the main constituent of the Arizona dust) was added to the resonator surface. Its thickness was varied to emulated PM2.5, PM5 and PM10 particulates and the sensor was simulated first in stationary state studies to compute the final temperature for each thickness setting. Since the resonator temperature was monitored while the model solver was running, this meant the transient studies that were run next could be stopped short once a sufficient approximation of the final temperature is reached, reducing computation time considerably. The result of the transient studies is shown in Fig. 4.9.
The thermal time constants for each setting were then calculated from the transient results. They can be read from Fig. 4.8 to be 0.95 s without the PM layer and to then increase to 1.00 s with a layer of PM2.5 added. PM5 increased this to approximately 1.01 s and PM10 increased it to 1.02 s. This behaviour is to be expected, since the addition of the PM layer increases the thermal mass of the device proportionally to the added size and hence also mass, too. In practice, the drop in steady-state temperature of the sensor is likely to be less than the 278 K shown in Fig. 4.8 since the sensor will not be covered by particles as densely as a solid layer of quartz does. However, calculating the effect of spherical approximations of randomly spread particles on the whole sensor substrate was too computationally intense for the hardware available at the time and so attempts to achieve this were abandoned. It was decided that practical tests would be needed to assess whether the response of the system as a particle sensor can be altered by the addition of particles. However, some frequency domain studies of particle deposition on a reduced model are presented in section 4.1.4.

As the sensor would not be back etched, the heat loss into the PCB needs to be considered prior to manufacture, too. Because of this a 1.6 mm thick, 2 cm × 2 cm block of FR4 was added to
the three-dimensional model [125][125]. Figure 4.10 shows the result of a steady-state study with a heater current of 60 mA including the PCB.

Fig. 4.10. SMR Temperature Including PCB (60 mA Heater Current) [125][125].

The total input power in Fig. 4.9 is approximately 220 mW, yielding a reduced maximum SMR surface temperature of 356 K. A transient study found the thermal time constant of the SMR on the PCB to be approximately 1.97 s, an increase of almost double compared to the SMR without the PCB. The addition of the PCB is also likely to reduce the effect of particles on the sensor temperature since this way they will cause a relatively lower change in the mass of the full system. Nevertheless, according to the model with the PCB, the power requirements of the design are also fulfilled.

Following the simulations above, the sensor design was then finalised and Cadence IC6® was used to draw the layout of the sensor for mask manufacture. The full sensor layout is shown in Fig. 4.11.
The Bragg reflector was sized 500 µm x 500 µm (only large enough to extend over the active area to reduce the heat loss through the metal layers). The minimum width of each chip for the available dicing was 1 mm, while the pads needed to be sufficiently far from the active area so that wirebonds do not obstruct particles and so that polymers or adhesive layers for particle or VOC capture may be added later, without accidentally spreading them onto the bond pads. Thus, the chip size was set to a length of 2.5 mm. The minimum pad size for the available wire-bonder was 50 µm. Because the sensor will be exposed to compressed air frequently it was decided to push the pad size to 150 µm x 150 µm, which will make it easier to repair broken wirebonds if required. A set of secondary pads was added to enable a secondary connection to the heater, for example for reference measurements in future. These were not used in this thesis.

### 4.1.3 3D Frequency Domain Simulations of SMR

With the sensor design set to that in Fig. 4.11, additional frequency domain simulations were carried out on the three-dimensional model to verify the resonant behaviour of the final device. This was also done to confirm if the temperature coefficient of frequency (TCF) would be practical and to investigate whether small particles would have an observable effect on the resonant frequency after deposition.

The two-dimensional COMSOL model assumed, as is commonly done, zero strain out of the model plane (plane-strain assumption) [10]. To obtain a better estimate of the CMOS SMR TCF the temperature coefficients of the relevant properties of the AlN were added to the three-dimensional model in Fig. 4.8. It was then simulated under 1 W perturbations across a wide
frequency range. The resonance region of the model’s S21-parameter is plotted against frequency in Fig. 4.12.

![Graph of S21 parameter at different temperatures](image)

**Fig. 4.12.** S21-parameter at Different Temperatures (COMSOL) [18].

The resonant frequency of the three-dimensional model is approximately 2.2 GHz and thus 10% higher than that of the two-dimensional model in section 4.1.1. S21 is also approximately 1 dB lower. Both can be explained by model differences, especially the lower resonance is most likely caused by lateral acoustic losses not accounted for under the plane-strain assumption. A TCF of –6 ppm/K can be calculated from this. This is likely a significant underestimation, since the TCF of AlN FBAR devices is typically around -25 ppm/K [182]. Variations are to be expected, as previous works using COMSOL have also reported significant differences between calculated and measured TCF values [10], [94]. One possible reason could be that FBAR devices are generally more temperature sensitive than SMRs [182]. Section 4.2.2 discusses the temperature coefficient from practical measurements in more detail.

To simulate successful particle deposition on the resonator’s top electrode surface, the model was reduced to the Bragg reflector area of the device only. Perfectly matched layers were used to
emulate the waves fading into the rest of the substrate. This reduced the number of mesh elements and thus computational effort. Initially the resonator’s mass sensitivity to Arizona dust was calculated by adding spheres of quartz. Their size was calculated using COMSOL’s integrated pseudorandom number (PSRN) generator with the same size characteristics as the “Ultrafine” Arizona dust of type ISO 12103-1 A1 proposed for experimental particle testing. The maximum particle size was set to 20 µm and the mean size to approximately 5 µm, with a normal distribution around the mean used for the PSRN generator. The particle position on the sensor surface was also calculated with the pseudorandom number generator to simulate the effect of random particle settling. Figure 4.13 shows a photo of the reduced model with particles shown in blue.

![Fig. 4.13. COMSOL Model of SMR Active Area with Spherical Particles (Position 1).](image)

Frequency domain studies were then run for different particle mass densities (1100 kg/m³, 2200 kg/m³ (actual quartz) and 4400 kg/m³) to confirm that the resonant frequency decreases proportionally to particle mass. Fig. 4.14 shows the result of this simulation.
Fig. 4.14. $S21$ vs. Frequency after Particle Deposition for Different Particle Densities.

With the density of quartz (2200 kg/m$^3$) the theoretical mass sensitivity of the SMR to particles is approximately 1 kHz/ng. $S21$ also changes by approximately 0.4 dB after particle deposition. This confirms that increasing particle mass increases resonator damping.

The sensitivity of resonator sensors is also dependent on the particle position. To demonstrate this, the seed of the random number generator for the particle position was then varied between different values. The different particle positions are shown in Fig. 4.15, with position 1 shown in Fig. 4.13 and the resulting frequency spectrum is shown in Fig. 4.16.
As was expected based on the results of previous works ([4], [94], [107], [109]) it can be seen from Fig 4.16 that the resonant frequency decreases significantly less (approximately 200 kHz) for the third particle position model compared to the first one. In the third model the number of large particles in the centre of the active area is lower, see Fig. 4.15. However, it should be noted that the assumption of full particle bonding to the resonator surface at the contact points probably
amplifies the effect that the particle position had on the resonant frequency in the simulations. In practical tests the particle bonding will be weaker and hence the effect of particle position might be smaller.

The effect of particle size was also investigated. For this the mean particle size of the third particle position (right-hand side in Fig 4.14) was varied in COMSOL between 2.5 µm, 5 µm and 10 µm. Figure 4.17 shows the result from this simulation.

![Graph showing S21 vs. Frequency for Different Particle Sizes.](image)

**Fig. 4.17. S21 vs. Frequency for Different Particle Sizes.**

Based on this simulation the 2.5 µm particles shift the resonant frequency approximately 75 kHz less than the 10 µm particles and 10 kHz less than the 5 µm ones. Additionally, the particle size influences signal damping, except for a spurious resonance at approximately 2258.6 MHz for PM10 where the sensor response decreases with increasing particle size. For example, the 2.5 µm particle simulation resulted in a S21 parameter that is 0.2 dB different from the 5 µm setting. The response is generally smoother when the particles are smaller, too. Based on this result it is likely that thermal expansion of the particles caused by sensor heating would increase the observed shift in resonant frequency and thus contribute to an increased particle sensitivity of the SMR. Compared
to Fig. 4.14 the particle size and resulting change in mass affects the response less than a change in mass of the same particles alone. In the simulations the particle position with constant particle distribution also affected the frequency shift more than the size did.

4.1.4 Design Summary and Layout for Mask Manufacture

Section 4.1 has described the design process of a custom CMOS SMR with an integrated microheater for particle sensing. It uses a combined 180 nm CMOS-BAW process offered by SilTerra Malaysia and will be manufactured by the same company. As using a standard CMOS process involves a trade-off between cheaper fabrication and ease of integration at the expense of a less optimised Bragg reflector stack, extensive verification of resonator performance was conducted prior to physical prototyping. The sensor was first simulated under electrode voltage perturbations to assess its resonance characteristics. Its theoretical resonant frequency was estimated at about 2 GHz. Following this, the thermal behaviour of the SMR was studied when a current was applied to its integrated microheater in steady-state and transient studies. The thermal time constant of the sensor on a PCB was found to be 0.97 s. Three-dimensional frequency domain studies were used to estimate the theoretical particle sensitivity of the sensor to be approximately 1 kHz/ng. The final sensor design measures 1 mm × 2.5 mm. Figure 4.18 shows the design layout submitted for manufacture.

Fig. 4.18 Die Layout Cells for Fabrication.

It measures 24.8 mm × 8.71 mm and consists of multiple sensor designs based on the layout in Fig. 4.10. They include different sizes of substrates and Bragg reflectors, as well as chips comprising multiple SMRs on the same die. Chips with non-parallel electrode edges or spiral rather than meander heater designs with and without rectangular corners were also manufactured.
Additionally, some of the sensors have exposed top-electrodes and some include a nitride passivation layer to protect the top electrode, for example from corrosion. Close-ups of the additional sensor layouts are shown in Appendix A. Following sensor manufacture, the electrode design with the best frequency performance was tested for proof-of-concept particle sensing detailed in chapter 6. An evaluation of alternative heater and Bragg reflector designs was beyond the scope of this research.

4.2 Fabrication of Thermally Modulated SMRs

The final design in Fig. 4.17 was then submitted to SiTerra Malaysia, who used it to prepare the wafer layout for mask manufacture. The full wafer map is shown in Fig. 4.19. 94 layout cells were fitted onto a 200 mm wafer layout, these dies are marked in green.

![Full Wafer Map](image)

**Fig. 4.19. Full Wafer Map (Image Credit: SiTerra Malaysia).**

In this case multilayer masks are used to reduce manufacturing costs. Four wafers of the sensors were fabricated in the SiTerra 180 nm CMOS-BAW process (described for 130 nm in [96], [97]) [125]. Section 4.2 will discuss the characterisation of the CMOS SMRs carried out by the author of this thesis following the receipt of the fabricated sensors at the University of Warwick.
4.2.1 Fabrication of SMR

Figure 4.20 shows a section of the CMOS SMR wafer after different manufacturing steps. The sensor the camera is focused on has a spiral heater design, see Appendix A for more details on this.
Fig. 4.20. Manufacturing Steps (Image Credit: SilTerra Malaysia).
In step 1 in Fig. 4.20 the microheater has been deposited on top of the bottom oxide layer. The pattern of the spiral design is well defined in the bottom metallisation layer. Each layer of the Bragg reflector stack is shown after deposition in steps 2, 3 and 4. Vias connecting the heater at the bottom to the bond pads in the top metal layer were also fabricated. The diamond-shaped via pattern connects the top layer of the bonding pads to the lower layer of the bonding pads. The top metal layer is also used to label each chip. After step 5 the top oxide layer of the reflector stack has been deposited. In step 6 the bottom resonator electrode is shown, and step 7 shows the wafer after the addition of the piezoelectric, the top electrode and the nitride passivation layer. Step 8 shows the wafer after the nitride and oxide have been etched away to expose the active area (on some designs) and the bond pads. In a final step the back of the wafer was ground down from 720 µm to 400 µm thickness. A photograph of a full wafer prior to dicing after receipt at the University of Warwick is shown in Fig. 4.21.

Fig. 4.21. Manufactured Wafer.

SilTerra Malaysia also diced one of the four wafers before it was shipped to the University of Warwick. This was done with a dicing saw, Fig. 4.22 shows dicing resulted in small fractures at
the chip edges, but these should not impact sensor performance, since the chip edges are at least 250 μm away from the edge of the Bragg reflector on all sides of the sensor.

![Sensor Dicing Damage](Image Credit: SilTerra Malaysia).

The sensors were thus ready to be picked off the wafer and tested. Section 4.2.2 will describe the physical device.

4.2.2 Manufactured SMR

Figure 4.23 shows a photograph of an individual chip of the custom-made CMOS SMR, while Fig. 4.24 shows a picture including approximate measurements of another chip taken using a Leica DM750M microscope with 5 × zoom and a Leica ICC50 W camera.
Fig. 4.23. Manufactured CMOS SMR [18].

Fig. 4.24. Leica ICC50 W Image of CMOS SMR.

It can be seen in Fig. 4.24 that the dice marks enclose an area of approximately 1 mm × 2.5 mm, while the Bragg reflector measures approximately 500 μm × 500 μm and the top electrode is
approximately 350 $\mu m \times 350 \mu m$ in size. A cross section of a sample of the chip, taken from near the centre of the wafer, photographed by a scanning electron microscope is shown in Fig. 4.25. It shown a well-defined layer stack as outlined in section 4.1. A close-up of the layer stack including the thickness of each layer in nanometers is shown in Fig. 4.26.

![SEM Cross-Section of Manufactured Device](image)

**Fig. 4.25.** *SEM Cross-Section of Manufactured Device* [18].
Figure 4.25 assumes the substrate has an accurate thickness of 400 µm. The design thickness for the CMOS layers was 530 nm for the metallisation layers and 850 nm for the oxide layers (top oxide 1 µm). Based on this, these layers deviate less than 4 % and less than 6 % from the design according to Fig. 4.25. The bottom electrode is within 6 % of the design thickness of 400 nm, while the piezoelectric layer has less than 4 % higher thickness. The surface of the piezoelectric can be seen to be slightly rougher than the other layers and this may have affected the deposition of the top electrode, which is approximately 16 % thicker than specified. The passivation stack has a thickness within 2 % of the specification. Even though these variations are mostly small, the manufactured SMR will likely have slightly different characteristics than the simulations in section 4.1 suggest. In the following, section 4.2.3 assesses the resonant and thermal behaviour of the CMOS SMR compared to the design simulations.

**Fig. 4.26. SEM Dimensions of SMR Layers (all in Nanometres).**
4.3 Frequency and Thermal Characterisation of SMR

To characterise the SMR, it was placed on a 2 cm × 2 cm FR4 PCB and attached using thermally conductive silicone adhesive paste. Its pads were wire bonded to connections for measurement instruments. The setup is shown in Fig. 4.26.

Fig. 4.27. SMR on PCB for Characterisation.

During the first set of tests only the microheater was tested to assess resonator temperatures, as well as the variation of the heater resistance with temperature. For this a Keithley 2602B Source Measure Unit (Source meter) was used to capture the variation of supply power and resistance as different heater currents are applied. The sensor surface temperature was measured with a FLIR SC7000 infrared camera [125]. An infrared image of the heated SMR with a current of 60 mA applied is shown in Fig. 4.28.
In Fig. 4.28 the sensor can be observed to heat evenly to approximately 345 K. The darker areas are areas with exposed metal pads for wire bonding or soldering and they thus have significantly lower emissivity than the sensor or the PCB, which the infrared camera has not been calibrated for [125]. This caused the temperature measurement to become inaccurate in these areas, so that the surface temperature of the device away from the electrodes was recorded for further analysis instead of the temperature of the active area. Apart from the exposed metal the temperature profile across both the device and the PCB looks very similar to that from the COMSOL simulation in Fig. 4.10. The steady-state results of this test have been recorded for 0 mA, 20 mA, 40 mA, 60 mA, 80 mA and 100 mA heater current in Table 4.2.
Table 4.2. *Heater Current vs Steady-State Resistance.*

<table>
<thead>
<tr>
<th>Heater Current (mA)</th>
<th>Heater Power (mW)</th>
<th>Surface Temperature (K)</th>
<th>Heater Resistance (Ω)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>286</td>
<td>48</td>
</tr>
<tr>
<td>20</td>
<td>20</td>
<td>295</td>
<td>49</td>
</tr>
<tr>
<td>40</td>
<td>80</td>
<td>306</td>
<td>53</td>
</tr>
<tr>
<td>60</td>
<td>205</td>
<td>345</td>
<td>61</td>
</tr>
<tr>
<td>80</td>
<td>512</td>
<td>413</td>
<td>81</td>
</tr>
<tr>
<td>100</td>
<td>1100</td>
<td>534</td>
<td>110</td>
</tr>
</tbody>
</table>

With no current applied to the microheater its resistance is approximately 48 Ω, which is within approximately 8% of the design value. A consequence of this is that measured temperatures will be lower than simulated ones.

**Fig. 4.29. Microheater Resistance vs Temperature.**
Using the data from Table 4.2, the temperature coefficient of resistance (TCR) was calculated for the microheater according to (4.10).

\[
TCR = \frac{R_2 - R_1}{R_1(T_2 - T_1)}
\]  

(4.10)

In (4.10) \(R_2\) is the resistance of the microheater at temperature \(T_2\), while \(R_1\) is its resistance at temperature \(T_1\). Assuming \(R_1\) and \(T_1\) are the values recorded at 0 mA heater current, an average TCR of 0.0045 ppm was thus calculated for the collected data. This value is approximately 13% higher than the 0.004 ppm TCR assumed in the simulations in section 4.1. One explanation for this difference is that the microheater temperature will be slightly higher than the SMR surface temperature used in (4.10). As it can be assumed that the heater was at room temperature when no current was applied, an underestimation of the heated temperature together with accurate resistance measurements would logically cause overestimation of the TCR of the microheater.

Table 4.3 shows the discrepancy between the surface temperatures measured on the SMR using the FLIR SC7000 compared to the temperature obtained at approximately the same point on the COMSOL model [125].

<table>
<thead>
<tr>
<th>Heater Current (mA)</th>
<th>Temperature COMSOL (K)</th>
<th>Temperature FLIR (K)</th>
<th>Discrepancy (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>295</td>
<td>295</td>
<td>0</td>
</tr>
<tr>
<td>40</td>
<td>315</td>
<td>306</td>
<td>2.9</td>
</tr>
<tr>
<td>60</td>
<td>354</td>
<td>345</td>
<td>2.6</td>
</tr>
<tr>
<td>80</td>
<td>436</td>
<td>413</td>
<td>5.6</td>
</tr>
<tr>
<td>100</td>
<td>562</td>
<td>534</td>
<td>5.2</td>
</tr>
</tbody>
</table>

As expected, the measured temperatures are lower than the simulations suggested they would be at each heater current. The average difference between the theoretical and practical sensor temperatures is approximately 3.3%, the maximum discrepancy measured is approximately 5.6%. Other explanations for the difference include the calibration of the FLIR SC7000 or difference in material properties between the COMSOL simulations and the manufactured device [125]. The sensor was also not inside a sealed chamber during these tests which means there could have been
some forced convection heat loss on the sensor surface that the simulations did not account for [125].

At a current of 125 mA the sensor failed. At this current the FLIR SC7000 recorded a maximum surface temperature of approximately 603 K [125]. A picture of the burnt sensor next to an intact one is shown in Fig. 4.30.

![Fig. 4.30. Top View of Overheated SMR (Top) and Unused SMR (Bottom).](image)

The top electrode has clearly deformed permanently and the whole chip has visibly charred across the top layer, which reasserts the fact that the sensor heats uniformly. This also supports the expectation from the simulations that thermal conduction is the dominant form of heat loss. The aluminium wire-bonds did not break from the overheating of the device.

To estimate the thermal time constant of the chip, the transient change in heater resistance can be used. A plot of the transient change in heater resistance after different currents are applied is shown in Fig. 4.31.
Fig. 4.31. Transient Change in Heater Resistance.

The crosses in Fig. 4.29 mark two-thirds of the final resistance value and hence the corresponding value on the x-axis is the thermal time constant measured in each of these tests. This is approximately 1.71 s on average, although this is for the chip on a PCB. Compared to the simulated thermal time constant of the chip only, which was approximately 0.95 s, this is approximately a 76% increase.

As it is relevant to changes in surface conditions, the FLIR SC7000 was used to record the transient change in SMR surface temperature while mounted onto a PCB, too. Figure 4.32 shows this together with the transient change in surface temperature calculated for the 3D model including the PCB.
The thermal time constant from the practical test is approximately 10% under the value from the simulation (1.85 s compared to 1.97 s) [18]. This is an increase of 8% compared to the thermal time constant calculated from the transient change in heater resistance. Since there would be a delay between the application of the heater current and the propagation of the thermal energy to the sensor surface, this observed difference in heating speed makes sense.

To characterise the behaviour of the piezoelectric resonator, the PCB from the microheater test was connected to a PicoVNA106 and the S21 parameter of the sensor was measured. The same currents as in Table 4.3 were applied. A 10 MHz spectrum around the peak in S21 is shown in Fig. 4.33.
Fig. 4.33. $S_{21}$ Parameter of the SMR with Different Currents Applied to the Heater [125].

At 0 mA current (room temperature) the theoretical resonant frequency of the chip was approximately 2,004 MHz from the 2D model and 2,213 MHz from the 3D model. This is approximately 1.4 % (28 MHz) or 10.7 % (217 MHz) higher than the measured resonant frequency of 1,976 MHz respectively. Considering the fabricated piezoelectric layer is 4 % thicker than the designed value this was to be expected. For example, when using (3.7) to estimate the resonant frequency of the resonator part of the chip only, the measured increase in piezoelectric layer thickness also leads to an increase in resonant frequency of approximately 3.8 %.

The value of $S_{21}$ at zero heater current is approximately -6 dB, which is 25% or about 12.5 % lower than expected from the 2D and 3D simulations respectively. According to (4.11) a quality factor of approximately 200 can be calculated from Fig. 4.33 [125].

$$Q = \frac{f_{S_{21}}}{\Delta f_{3dB}}$$  \hspace{2cm} (4.11)

The resonant frequency, $S_{21}$ and the Q factor decrease (resonator damping increases) as the heater current increases, as is expected to happen with increasing temperature.
To estimate the TCF of the CMOS SMR it was connected to an E5071B vector network analyser (VNA) by Agilent Technologies to continuously record the resonant frequency. It was then placed in a sealed zero air chamber inside a controlled temperature oven, the temperature of which was varied in 5 K steps between 303 K and 353 K. Figure 4.34 shows the measured resonant frequency and temperature over time and Fig. 4.35 shows the average resonant frequency at each temperature setting used to calculate the TCF.

![Graph showing resonant frequency and temperature variation](image)

**Fig. 4.34.** Resonant Frequency Variation as Temperature is Ramped Up and Down.
Using the data from Fig. 4.35 a TCF of -27 ppm was estimated for the CMOS SMR, which is approximately that expected for AlN resonators [182]. As expected, this is larger than the simulated value, most likely because of differences between the temperature dependence of theoretical and practical material properties, for example of the stiffness of each layer. The effect of temperature on ambient air pressure is not accounted for in the model and might have an effect. Differences in TCF can also arise because of variability in the material crystal formation during manufacture. Different crystal types can have different coefficients of thermal expansion, which affects thermal stresses inside the device, as well as changes to its dimensions [10]. The model does not take these into account.

4.3.1 SMR within an Oscillator Circuit
For signal read-out the SMR will be used in a Pierce oscillator circuit. Since it will form the frequency-setting element its characteristics have a direct impact on circuit performance. This section will briefly cover the sensor’s performance when in a Pierce oscillator. The sensor was wire bonded to a Pierce Oscillator PCB designed by Dr Siavash Esfahani at the University of Warwick. A circuit diagram of this circuit that is based on that by Thomas et al. [12] is shown in Fig. 4.36.

![Fig. 4.35. Resonant Frequency vs Temperature.](image)
Here Q1 is the amplifying element, R1 and R2 are the biasing resistors and C1 and C2 are the frequency selecting capacitors from Fig. 3.1. L1 was added for biasing of the output. The output frequency spectrum of this circuit then was recorded with a Tektronix MDO3012 spectrum analyser and the result of this is shown in Fig. 4.37.
The frequency of oscillation of the circuit is approximately 1.4 GHz, which is lower than the resonant frequency of the SMR. This can be explained by the size of the load capacitors used in the feedback loop [125]. Because of time constraints these were not optimised further for the new SMRs and were deemed sufficient for further testing.

Fig. 4.38 shows the normalised resonant frequency of the oscillator recorded over time when currents of 40 mA and 60 mA are applied to the microheater and then removed after the signal has stabilised [125].
This test demonstrates a strong dependence of the oscillator’s output frequency on the SMR temperature [125]. For example, the output frequency of the circuit changes by approximately 1.75 MHz when a current of 60 mA is applied to the microheater, which corresponds to a SMR temperature change of approximately 318 K [125]. The settling time of the signal is approximately 5 minutes [125], which means temperature modulation with this circuit will have to be applied at a significantly slower rate than the chip’s thermal time constant would allow.

4.4 Chapter 4 Conclusions
This chapter has presented the design, and simulation of a novel CMOS compatible SMR sensor with temperature control through an integrated microheater. The sensor was fabricated closely to the design specification by SilTerra Malaysia in a fully integrated CMOS-BAW technology illustrated in section 4.2.1. In performance characterisation tests the CMOS SMR performed approximately as expected. The SMR was shown to function as the frequency setting element in a discrete Pierce oscillator circuit, with direct frequency control possible through temperature modulation of the sensor. To establish the capability of the CMOS SMR as a particle sensor and to assess whether thermal modulation of the device can increase the sensor’s performance further,
separate tests were carried out using a custom-built particle test rig. The results of these tests will be presented and discussed in Chapter 6, while Chapter 5 will focus on the construction of the particulate measurement test rig and the experimental evaluation of a thermophoretic sampling channel to be used with the novel SMRs.
5 Thermophoretic Particle Deposition

Chapter 4 described the design, simulation, and characterisation of a CMOS SMR for particle sensing with an integrated microheater. The time prior to the receipt of the fabricated sensor at the University of Warwick was used to set up the test rig required to test the SMR’s capability as a particle sensor. In section 2.1 and 2.2 it was outlined how the sensitivity of MEMS particle sensors as well as the repeatability of measurements for aerosolised particles is currently their main limitation. To test the repeatability of sensor measurements a test rig comprising a computer-controlled aerosol generator feeding into a sealed sensor test chamber with integrated sensor control was assembled. During fabrication of the sensor presented in chapter 4, this test rig was used to investigate whether the particle sensing capability of an existing FBAR device supplied by SilTerra Malaysia can be improved by the addition of a thermophoretic particle deposition mechanism using a microhotplate array provided by the Materials Center Leoben, Austria (MCL). This chapter will first present the design and functionality of the test rig and then evaluate the FBAR particle sensor with and without the addition of external particle sampling mechanisms.

5.1 Design of a Test Rig for Different Pollution Levels

This section focuses on the test setup for the MEMS particle sensors. It comprised an aerosol generator that injected a mix of particles into a stream of compressed zero air, which was then fed into a glass test chamber. The aerosol generator used was a Topas SAG 410/L [231]. The rate at which particles were injected into the air stream could be altered and hence remote control of the pollution level inside the chamber was possible. Figure 5.1 shows a schematic drawing of this setup.
Fig. 5.1. *Particle Test Rig Schematic Drawing* [18].

Commercial sensors were used as references to monitor the pollution level inside the chamber. These were an Alphasense OPC-N2 and a Sensirion SPS30 [82], [232], [233]. A Bosch BME280 was used to monitor the temperature, pressure, and humidity levels inside the chamber [234]. A National Instruments® (NI) USB 6009 data acquisition device (DAQ) was used to control the aerosol generator and an Arduino Due microcontroller was used to control the commercial sensors. A custom interface was created in NI LabView to send and receive commands to and from the DAQ and the Arduino. The PCBs with the drive and readout circuitry for the microhotplate array and the FBAR particle sensors were controlled by a Teensy 3.6. The LabView interface was also used to send control commands to the Teensy 3.6 and to receive and plot the measured frequency over time. The outlet air stream was fed through a bubbler and a high-efficiency particulate air (HEPA) filter into a ventilated fume cupboard. Sections 5.1.1 and 5.1.2 will describe the individual parts of the test rig in more detail.

5.1.1 Test Chamber
Several considerations were important for the design of the test chamber in Fig. 5.1. Most importantly it needed to be capable of accommodating all the sensing devices and enable their connection to the control PC, while remaining completely sealed during the tests. The latter was
important to ensure no air or particles could escape from the chamber unfiltered, because of the health hazard to the rig operator posed by aerosolised particulate matter. Thus, the chamber needed to be made from a non-porous material. It was also desired that the particle test chamber should be clear, to enable good visual observation of the experiments. Because of this glass was chosen as the material for the test chamber. An alternative would have been acrylic, but glass has higher rigidity, which should reduce pressure variations inside the chamber. Glass also does not accumulate static charge as easily as acrylic does, which leads to less attraction of particles by the chamber itself. Furthermore, glass is more scratch resistant, which was useful during regeneration of the rig between tests.

The test chamber was designed to have a size of 0.6 m × 0.6 m × 1.0 m. The size was chosen to provide sufficient space for a range of devices, including the possible future addition of more bulky reference particle monitors. The input gauge air pressure from the aerosol generator in Fig. 5.1 is 1 bar. COMSOL Multiphysics was used to confirm a size of 12 mm for air inlet and outlet would avoid pressure build-up inside the chamber. Air inlet and outlet were placed centrally at the top and the bottom of the chamber because this should cause greater air turbulence in the chamber and hence help to distribute the particles evenly. The air flow velocity field calculated in this simulation is shown in Fig. 5.2.
Fig. 5.2. *Air Flow Streamlines Inside Test Chamber (COMSOL).*

According to this result, air flow is present throughout the chamber, as desired. Following this, holes were added to the front and back panel design of the chamber for the air inlet and outlet and for the cables to connect and power the instrumentation that would be placed inside later. The glass chamber was manufactured to this specification by ND Aquatics, Cambridge, UK. A picture of the manufactured test chamber with additional IP68-rated (EN 60529) KVT-ER cable glands by Icotek, Eschach, Germany for the connection cables and EXCGM cable glands for the air line by Kopex, Birmingham, United Kingdom, attached to the holes is shown in Fig. 5.3.
The inside of the chamber is accessible through an opening at the top. As was highlighted in section 2.1, PM is hazardous and thus so is the aerosolised test dust, which meant the test chamber required careful sealing and filtration of the polluted air output. With the assistance of Mr Frank Courtney at the University of Warwick, a glass sheet was attached to an acrylic frame, which could be lifted off the top of the chamber for access. Metal clamps were used to affix the lid onto the chamber during tests, this was sealed with IP68-rated rubber strips. Figure 5.4 shows an image of the sealed test chamber including the clamped top lid.
For additional safety from accidental sudden pressure increases, a 2-bar emergency pressure relief valve was added. The output was initially filtered using a water bubbler only. Both the bubbler and the valve are visible at the bottom right of Fig. 5.4. The setup was then tested with a fog machine, and it was found that, while the chamber was fully sealed with no leakage, some vapour escaped through the bubbler. For additional filtration of the output air, a H14 HEPA filter was added to the output air pipe, following the bubbler. This filter was then placed inside an externally ventilated fume cupboard to extract the output airstream safely. As an additional precaution protective particle-filtering face masks certified in Europe according to the EN149 standard as Filtering face Pieces (FFP) 3 [235], as well as latex gloves, safety goggles and laboratory coats were worn during all experiments and whenever the chamber was opened.

Between experiments the regeneration of the setup, i.e. rig cleaning, and hence the removal of settled aerosol particles from the rig was required. To ensure particles remained captured after
removal from the chamber, the test equipment was cleaned after each test with a Numatic CRQ370-2 clean room vacuum cleaner. Additionally, the sensors were cleaned using compressed air.

5.1.2 Aerosol Generator and Commercial Reference Sensors
To ensure accurate control of the particle mass flow rate into the test chamber, the Topas SAG 410/L aerosol generator was used [4]. It comprises a dosing belt to deliver a consistent mass of particles to an injection nozzle [231] according to the German engineering standard VDI 3491 [236]. This principle of aerosol generation thus uses the venturi effect, it follows to the international standard ISO 5011 [231], [237]. An image of the device is shown in Fig. 5.5.

![Fig. 5.5. SAG410/L Connected to DAQ for Remote Control.](image)

Inside the SAG410/L the inlet airstream passes through a solenoid valve into a venturi tube (a nozzle followed a diffusor). The feeding belt inside the SAG410/L comprises uniformly sized compartments. These are filled from the particle reservoir by a scraper to ensure consistent filling. Control of the belt speed enables control of the rate at which each of the filled compartments
reaches an inlet between the nozzle and the diffusor. The low pressure at the outlet of the nozzle causes a suction effect through which the particles at the inlet are injected into the airstream through the diffusor [238].

To enable remote control of the valve and the belt speed, the SAG410/L was connected to a NI USB-6009 DAQ via a 15-position D-SUB connector. The DAQ was controlled through a LabView interface programmed for this purpose. This is shown in Fig. 5.6.

![LabView Control Interface for SAG410/L](image)

**Fig. 5.6. LabView Control Interface for SAG410/L.**

The DAQ provided a voltage between 0 V and 5 V, set on a dial in the control interface, to the feeding belt’s electric motor. The mixed size particle airstream was then passed through the diffusor and directed into the test chamber at a gauge pressure of 1 bar. The chamber thus had a variable level of aerosolised particle concentration, with a reliably calculable particle feed rate per unit volume of the test chamber.

The test dust used for the experiments in this research was Arizona dust of type ISO 12103-1 A1 “Ultrafine. Quartz is the main constituent of the test dust (> 90 %). Figure 5.7 shows an image of the test chamber with diffused aerosolised particles and a uniformly spread layer of settled particles at the bottom.
Fig. 5.7. Test Chamber with Aerosolised Particles.

The particle concentration inside the chamber was measured using a Sensirion SPS30 and an Alphasense OPC-N2. These are both optical particle counters. They were chosen primarily because of their low cost and small size and because the OPC N-2, for example is used extensively by the US EPA. Both sensors have limited accuracy [58], [80], but they were found to be able to give a reliable indication of the relative pollution level inside the chamber over time. Especially the OPC-N2 could differentiate the level of pollution even at the higher particle concentration settings used during the experimental tests, where the SPS30 was found to saturate. An Arduino Due microcontroller was programmed to receive control commands for the sensors from the LabView interface to pass on to the sensors for data readout and to then transmit the measurement data back to the control PC for storage. The Arduino Due also controlled the Bosch BME280 for temperature, humidity, and pressure measurement. It was sealed inside the chamber in a metal box and connected to the PC via universal serial bus (USB). The reference sensor control interface is
shown in Fig. 5.8. The data from these commercial reference sensors was plotted in real time. This enabled close monitoring of chamber temperature, pressure, and humidity stability.

**Fig. 5.8. LabView Control Interface for Reference Sensors.**

Figure 5.9 shows the PM10 particle concentration inside the test chamber measured with the OPC-N2 for different feeding belt settings. In these tests the feeding belt speed was set to 0.25 V, 0.5 V, 0.75 V and 1 V and the particle feeding was enabled for 5 minutes [4]. The black line in Fig. 5.9 indicates the time at which the particle injection into the airstream was disabled. To reduce noise the data was filtered with a 20th order median filter.
It was calculated from the SAG410/L datasheet that the rate of increase in particle concentration inside the test chamber for 0.25 V, 0.5 V, 0.75 V and 1 V feeding belt speed setting was 19 µg/m³s⁻¹, 43 µg/m³s⁻¹, 66 µg/m³s⁻¹ and 94 µg/m³s⁻¹, respectively [4]. The shapes of the plots in Fig. 5.9 show that the OPC-N2 could measure a faster rise in particle concentration when the particle feed rate was increased. The particle concentration rises very quickly while particles are fed into the chamber. For each test the concentration plot reaches its maximum at around 5 minutes, confirming a consistent particle feed rate. This implies that doubling the feeding belt speed approximately doubled the peak particle concentration as well. Additionally, after the feeding belt is switched off, the concentration declines gradually and approaches zero after approximately half an hour for each of the tests. This is because some particles remain aerosolised inside the chamber for some time before settling at its base or leaving through the outlet. This meant that the minimum time particle tests would have to run for was thirty minutes, to ensure the level of airborne particles inside the chamber returned to a safe level before opening it for sensor regeneration and rig cleaning. As it will be explained in section 5.2, this time was further increased by the need to wait for sensor base line settling time prior to each particle test.
The maximum particle concentrations reached at the feed settings tested in Fig. 5.9 were, according to the OPC-N2, approximately 1000 µg/m³, 2000 µg/m³, 3000 µg/m³, and 4000 µg/m³. One limitation of the test rig is hence that these values are substantially higher than the ambient particle concentrations of interest for commercial urban air quality monitoring (closer to 40 µg/m³ in the UK for example [239]) [4]. Nevertheless, these values were used for the particle testing in this research for the proof-of-concept because a device capable of diluting the feed rate further was not available. More importantly, as was explained in section 2.1.6, FBAR particle sensors currently have limited particle sensitivity. It was observed during initial test runs that these concentrations would be suitable for the tests of the particle sampling and sensing principles described in the following chapters.

5.1.3 Full Setup and LabView Control
A photograph of the completed and sealed test rig is shown in Fig. 5.10. It was assembled and stationed in a temperature-controlled room with air conditioning.
An aluminium structure with wooden shelves was used to provide support for the instrumentation at the appropriate places for each part. A monitor for the LabView control interface was attached to the rig, see Fig. 5.10. The output HEPA Filter is shown inside a fume cupboard and connected to the bubbler at the chamber’s air outlet.

The printed circuit board (PCB) stack bearing the BAW sensors investigated in this work were positioned at the centre of the rig. For data readout a Teensy 3.6 was plugged into this stack and a USB cable was used to connect it to the drive PCB through the sealed cable glands shown in Fig. 5.3. A metal vice with insulated jaws was attached to the bottom of the glass chamber. These were used to reposition the sensor PCBs consistently after each time they were removed from the chamber for sensor cleaning. The differential sensor frequency data was also acquired and stored through a LabView interface, as shown in Fig. 5.11.

This interface was also used to control the current through the microhotplates for thermophoretic deposition, as well as their switching frequency, the particle sampling fan speed and to control the sensor temperature and switching period once the new devices with the integrated microheater were used.

The LabView interfaces in Fig. 5.6, 5.8 and 5.11 were combined into a single LabView interface shown in Fig. 5.12.

**Fig. 5.11. Control Interface for Microhotplates and Temperature Modulation.**
Fig. 5.12. *Particle Test Rig LabView Control Interface for Two Sets of BAW Devices.*

The data acquired from the BAW particle sensors inside the particle test rig was plotted on this interface in real time during the tests.

After completion of the test rig described in this section, experimental tests were carried out with it. As was mentioned in the introduction of this chapter, these initially included the evaluation of an FBAR device supplied by SilTerra (SilTerra FBAR) to assess its capability to be used as a particle sensor. Section 5.2 will discuss the results of these experiments.
5.2 FBAR Particle Sensor with Thermophoretic Deposition

As was explained in section 2.3, the functionality of MEMS particulate matter sensors can be improved with additional particle sampling and deposition mechanisms. This is because more efficient capture of aerosolised particles on the resonator surface results in a larger measurable frequency shift at the same particle concentration. Thus, the sensitivity of the sensor to aerosols is effectively increased without needing to improve the sensor itself. In this section a SilTerra FBAR is used to investigate how the addition of thermophoretic particle deposition can improve its capability as a particle sensor. First the setup is described and then the results from experimental tests using the test rig described in section 5.1 are evaluated. This work was published in [4].

5.2.1 MCL Microhotplates for Thermophoresis

The thermophoretic effect can alter the path of aerosol particles through the difference in the kinetic energy of the air molecules surrounding them created through a temperature differential [4]. Particles may thus be guided onto the surface of a MEMS particle sensor from an airstream by heating the air above the particles [4]. An advantage of this method is that the sensor does not have to be located directly in the sampling airstream and is thus less dependent on its properties. Thermophoresis also has high potential for particle type/size separation within a single sampling channel by using a range of temperature differentials [4]. An array of eight microhotplates supplied by the Materials Centre Leoben, Austria was used to induce the thermophoretic effect in this work. The array was originally developed for chemical sensing [4]. A photograph of the microhotplate array is shown in Fig. 5.13 [4].
The microhotplate array was fabricated by AMS AG, Austria, in a 350 nm CMOS process [4]. Polysilicon was used as the heater material for the microhotplates [4]. In the first fabrication step they were etched out of the CMOS stack on top of the silicon wafer. Next a titanium layer with 5 nm thickness and a platinum layer with 150 nm thickness were deposited [4]. The electrodes were patterned on the microhotplates in a lift-off process [4]. In a dicing step the wafer was cut into 2 cm × 2 cm chips and an SnO$_2$ film with 50 nm thickness was deposited on them [240], [4]. The SnO$_2$ films were patterned to cover only the microhotplates [4]. Additionally, a dry-etching process was used to under-etch the microhotplates [4]. Stealth dicing was used to cut the chips into single arrays with a size of 4.7 mm × 5.0 mm [4]. The microhotplates had a resistance of approximately 1 kΩ and could achieve a final temperature of approximately 673 K in 10 ms when 13 mW of power were supplied [241], [4].

Constant current sources according to a source follower configuration were used to power the microhotplates, see Fig. 5.14.
Fig. 5.14. Computer-controlled Current Source.

The heater current and hence temperature was set by altering the resistance of the potentiometer shown in Fig. 5.14.

The chip used for this was an Analog Devices AD5206 digital potentiometer. Its resistance was set using the Teensy 3.6. The Teensy was also used to control the MOSFET in Fig. 5.14 and hence its power state. The current and switching speed were set through the LabView interface shown in Fig. 5.11 and communicated through serial commands to the Teensy. Dr Siavash Esfahani designed a PCB on which the circuit in Fig 5.14 was implemented and replicated for each microhotplate using the multiple channels on the AD5206. This was used to drive the microhotplates for the experimental tests in this chapter.

5.2.2 SilTerra FBAR
As mentioned above, a FBAR device supplied by SilTerra was used to evaluate the thermophoretic sampling mechanism. A top-view photograph of this device is shown in Fig. 5.15 [4], while a schematic cross-section of the sensor is depicted in Fig. 5.16 [4].
The SilTerra FBAR was fabricated in their 130 nm CMOS-BAW process [4]. It uses aluminium nitride with a thickness of 1.3 μm as the piezoelectric material [4]. The top and bottom electrodes were made of aluminium with thicknesses of 400 nm and 350 nm respectively and with
7779 μm² of surface area [4]. A Si₃N₄ passivation layer covered the sensor to seal the air cavity and increase its ruggedness [4].

For particle sensing the FBAR was used as the frequency setting element in the Pierce oscillator circuit similar to that in Fig. 4.36. As mentioned previously, this circuit was adapted from a schematic design by Thomas et al. [12]. Only the load capacitors (C₁ and C₂ in Fig. 3.1) were changed to 0.5 pF to reflect the smaller capacitance of the SilTerra FBAR compared to the SMRs used by Thomas et al. Each Pierce oscillator resonated at approximately 1.1 GHz [4].

5.2.3 Thermophoretic Sampling Channel

As was described in section 3.2.2, the particle sensing experiments used the BAW sensors in a differential configuration for frequency readout. This principle is illustrated again in Fig. 5.17.

![Diagram](image)

**Fig. 5.17. Block Diagram of Differential Configuration.**

In this case the two oscillators were integrated on a single PCB designed by Dr Siavash Esfahani at the University of Warwick. The sensing oscillator was wire bonded onto the top of the PCB and the reference on the bottom [4]. The frequency difference between the two oscillators was passed to a Teensy 3.6 microcontroller using a Mini-Circuits RMS-30+ mixer circuit on a separate PCB. Because no particles could land on the surface of the reference FBAR the frequency differential contained information related to the effects that were not common mode and hence the shift in frequency could be related to the mass of particles captured on the sensing oscillator surface [4]. An ADCMP600 comparator was used to increase the logic level of the frequency differential and a Teensy 3.6 microcontroller was plugged into the mixer circuit PCB to measure its output frequency directly.

The microhotplates were placed above the sensing FBAR and a Sunon UB393-700 fan blower was used for air sampling [4]. A schematic drawing of this setup is shown in Fig. 5.18 [4].
A channel was 3D-printed to serve as a holding bracket for the microhotplate and the Pierce oscillator PCBs, which also formed its upper and lower wall [4]. A photograph of the thermophoretic particle sampling channel with the FBAR sensors and readout circuitry is shown in Fig. 5.19.
5.2.4 Experimental Procedure

The system in Fig. 5.19 was then tested inside the particle test rig described in section 5.1. The setup was first tested for particle sensing without the microhotplate PCB and the sampling fan (Method A), to be able to assess the performance improvement achieved by the addition of the sampling and deposition mechanisms [4]. Next the device was tested with the assembled channel and the microfan, but without switching the microhotplates on (Method B) [4]. Finally, a set of tests was executed employing both the sampling fan and the thermophoretic deposition mechanism (Method C) [4].

The tests for each of these three methods was repeated three times for five different particle concentration setting. As mentioned above, the speed setting of the SAG410/L is determined by its drive voltage [4]. During the tests the belt speed was set to voltages of 0.1, 0.25, 0.5, 0.75 and 1 V, which corresponded to 2, 5, 10, 15 and 20% of the full speed at 5 V [4]. This resulted in particle feed rates into the chamber of approximately 9.5, 19, 43, 66 and 94 µgm⁻³s⁻¹ or peak particle concentrations of approximately 500, 1000, 2000, 3000 and 4000 µg/m³ according to the OPC-N2 [4]. Between tests the chamber was regenerated as described in section 5.1.1. and the sampling
channel was cleaned using compressed air [4]. At the start of each test the recording of the output signal of the mixer circuit, as well as from the reference sensors was enabled. After this, the flow of the zero-air was turned on and the signal was observed until the pressure, humidity and temperature inside the chamber had stabilised [4].

The stable mixer output signal was recorded for thirty minutes before the particle injection into the stream of zero air was enabled [4]. It was disabled after five minutes, as described in section 5.1.2. [4]. The flow of zero air was disabled 40 minutes after the particle feed was enabled, to ensure the level of aerosolized particles had returned to a negligible level inside the test chamber [4]. The test results are presented in the following section.

5.2.5 Particle Sensing with and without Microchannel

The FBAR devices were directly exposed airflow in the chamber in the first set of experiments according to Method A. Figure 5.20 shows the normalised output frequency of the mixer circuit (the difference in resonant frequency between the sensing and the reference FBAR) [4].

Fig. 5.20. Measured Frequency Difference for Method A [4].
The data in Fig. 5.20 was measured with the Teensy 3.6 and filtered with a 250th order median filter to remove random noise from the signal [4]. It can be seen from Fig. 5.20 that the average normalised frequency difference for a particle feed rate of 9.5 $\mu$gm$^3$s$^{-1}$ shifts by approximately 3 kHz after the particle feed is enabled [4]. For 19 $\mu$gm$^3$s$^{-1}$ the shift is approximately 4 kHz and for 43 $\mu$gm$^3$s$^{-1}$ it is approximately 5 kHz [4]. The most noticeable shift in oscillator frequency difference occurs at the highest particle feed rates and hence the highest particle concentrations tested [4]. Because of the reliance on random particle settling on the FBAR surface according to Method A, the repeatability of the tests carried out in this experiment was poor [4]. This will be discussed in more detail in section 5.2.6.

A set of normalised results from the second type of test, according to Method B, is shown in Fig. 5.21 [4].

![Fig. 5.21. Measured Frequency Difference for Method B [4].](image)

These results were also filtered with the median filter used for Fig. 5.20. According to Fig. 5.21, adding the microchannel, as well as the sampling fan to the sensing FBAR increased the frequency shift observed at each particle concentration [4]. About 3 kHz of frequency shift were
observed for 9.5 \( \mu g/m^3/s \) particle feed rate, like for Method A. However, the frequency shift for 19 \( \mu g/m^3/s \) increased to approximately 15 kHz and to 30, 30 and 37 kHz for 43, 66 and 94 \( \mu g/m^3/s \), respectively, when using Method B [4]. An immediate occurrence of the frequency drop was also observed for these tests, while at 94 \( \mu g/m^3/s \) the drop only happens after approximately 5 minutes for method A [4]. It can also be seen from Fig. 5.21, especially at 19 \( \mu g/m^3/s \) particle feed rate, that approximately 15 minutes after the particle injection was disabled, the frequency measurements slowly began to return to the level they were on at the start of the test [4]. This suggests that the fan caused some degree of sensor cleaning once the concentration of aerosolised particles had dropped sufficiently [4].

In Method C the microhotplates inside the channel used for Method B were switched on and heated to approximately 673 K. This was to enable the thermophoretic precipitation of particles onto the resonator surface, which should increase the particle sampling efficiency of the particle sensing system, as well as increase the repeatability of the frequency measurements at each particle concentration. Figure 5.22 shows a set of results for FBAR particle sensing according to Method C [4].

![Fig. 5.22. Measured Frequency Difference for Method C [4].](image)
The frequency shift for a particle feed rate of 9.5 µg/m³s was increased by a factor of approximately 8 to 25 kHz. The increase was approximately double that observed using Method B for 19 µgm⁻³s⁻¹ (35 kHz instead of 15 kHz) [4]. In Fig. 5.21 and Fig. 5.22 the rate of frequency drop can also be observed to increase with increasing particle concentration [4]. For particle concentrations of 43, 66 and 94 µgm⁻³s⁻¹ the measured frequency shifts according to method C was also approximately doubled, to 55, 70 and 75 kHz, respectively [4]. Figure 5.23 shows how the frequency measurement drops as the particle concentration inside the chamber increases for this test (19 µgm⁻³s⁻¹ particle feed rate) [4].

![Figure 5.23](image)

**Fig. 5.23. Mixer Output vs Alphasense OPC-N2 Commercial Sensor** [4].

It can also be observed that the frequency returns to the baseline approximately 5 minutes after the particle feed is turned off. This reemphasises that there seems to be some sensor cleaning occurring under a certain threshold of particle concentration [4]. In addition, the return of the resonant frequency to the baseline after the particle injection was disabled is visible clearly in Fig. 5.22 for the three lowest particle concentration settings [4].
The test rig temperature and humidity were monitored during each experiment. Figure 5.24 shows the chamber temperature during the test in Fig. 5.23.

![Graph showing temperature and normalized frequency over time.](image)

**Fig. 5.24. Mixer Output vs Chamber Temperature.**

It can be seen in Fig. 5.24 that the temperature is stable with fluctuations within a range of less than 0.2 K. There is no visible correlation between the temperature and the shift in resonant frequency observed after the particle injection.

In Fig. 5.25 the relative humidity inside the test chamber is shown.
The humidity drifted upwards during the test. Based on Fig. 5.25, there was a total increase in humidity of approximately 0.5 %. Similar to the chamber temperature, this data also does not correlate with the shift in resonant frequency observed after particle injection. Random noise caused measurement fluctuations within a range of 5 kHz. There were no other known disturbances to the particle test rig for the duration of these experiments and hence it can be reliably assumed that the observed frequency shifts were caused by particle deposition only.

5.2.6 Improvement in Particle Sensor Performance
The results presented in section 5.2.5 show a clear improvement in the sensing performance of the FBAR particle sensor after the thermophoretic particle deposition mechanism was added. Figure 5.26 shows the resulting mixer output frequency shifts measured during the repeat tests for each tested particle concentration that were calculated as the difference between the average frequency measured before and after the particle injection of the chamber [4].
As expected, the output frequency of the mixer decreased with increasing particle concentration. Thus, as the top electrode captures particles, the resulting mass loading of the sensing FBARs led to a decrease in the resonant frequency of the FBAR and hence the oscillator circuit’s output frequency. This is consistent with the Sauerbrey equation. Equations (5.1), (5.2) and (5.3) provide the lines plotted in Fig. 5.26 that were fitted to the data from Method A, B and C, respectively [4]. They are based an empirical model of saturation (Langmuir isotherm). Especially the result from Method C suggests an asymptotic saturation of the FBAR’s ability to detect particles exists above a certain level of aerosolised PM [4]. In (5.1), (5.2) and (5.3) the variable $C$ represents the particle concentration that results in a corresponding shift in the measured frequency of the FBAR represented by $f_{shift}$ [4].

$$f_{shiftA} = \frac{1.5 \times 10^6 \times 3.5 \times 10^{-6} \times C}{1 + 3.5 \times 10^{-6} \times C}$$  \hspace{1cm} (5.1)$$

$$f_{shiftB} = \frac{9.1 \times 10^4 \times 1.7 \times 10^{-4} \times C}{1 + 1.7 \times 10^4 \times C}$$ \hspace{1cm} (5.2)
Based on (5.3), a shift in the resonant frequency of 5 kHz could thus be used to detect levels of particulate matter concentration as low as 50 µg/m³ in the test chamber [4]. The regulatory PM10 limit in the United Kingdom is 40 µg/m³ and thus not far off this theoretical detection limit [4]. Because the Sauerbrey equation suggests that the FBAR’s mass sensitivity may be increased by increasing the resonant frequency of the device, optimisations to the magnitude of the resonant frequency may improve the suitability of this particle sensing system sufficiently for real-time, in-situ monitoring of air quality [4]. As the SiTerra FBAR was not initially designed for use as a mass sensor, optimisation of its Q factor for sensing applications may also lead to some improvement [4]. Nevertheless, further tests are needed to confirm the minimum achievable level of reliable particle concentration measurement with this sensing system.

When comparing the data points in Fig. 5.26 for the different methods of particle sampling, there is an obvious improvement in the mass sensitivity of the sensing system [4]. For example, when relying on random particle settling, a particle mass concentration of approximately 4000 µg/m³ caused approximately 20 kHz of signal frequency shift [4]. Adding the microfan lead to a shift of 30 kHz at half the particle concentration (2000 µg/m³) using the same FBAR and circuitry [4]. Hence, as expected, it can be assumed that there was a significant increase in the efficiency of the particle deposition on the sensor when using the microchannel [4]. As shown in Fig. 5.26 the frequency shift increased even further, to approximately 55 kHz after the thermophoretic particle deposition device was added [4]. This reiterates the improvement achieved by adding the MCL microhotplates to the channel [4]. Based on these results the sensitivity of the system to aerosolised particulate matter increased from 15 to 27.5 Hz/µgm⁻³ between methods A and B [4]. Between methods A and C this value was improved from 5 to 20 Hz/µgm⁻³ for 4000 µg/m³ [4]. For a comparison of the results, the mean shift in the frequency measurement is shown in Table 5.1 for each test at each particle concentration tested.

\[
f_{shiftc} = \frac{9.9 \times 10^4 \times 6.8 \times 10^{-4} \times C}{1 + 6.8 \times 10^{-4} \times C}
\]
Table 5.1. Mean Frequency Shift Results in Fig. 5.26.

<table>
<thead>
<tr>
<th>Particle Concentration (µg/m³)</th>
<th>Frequency Shift Method A (kHz)</th>
<th>Frequency Shift Method B (kHz)</th>
<th>Frequency Shift Method C (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>5.3</td>
<td>2.9</td>
<td>28.1</td>
</tr>
<tr>
<td>1000</td>
<td>3.2</td>
<td>14.9</td>
<td>36.5</td>
</tr>
<tr>
<td>2000</td>
<td>6.3</td>
<td>26.1</td>
<td>56.1</td>
</tr>
<tr>
<td>3000</td>
<td>16.0</td>
<td>19.3</td>
<td>68.6</td>
</tr>
<tr>
<td>4000</td>
<td>21.4</td>
<td>37.6</td>
<td>74.8</td>
</tr>
<tr>
<td>Mean Shift (kHz)</td>
<td>10.4</td>
<td>20.1</td>
<td>52.8</td>
</tr>
</tbody>
</table>

By adding the microfan, the mean shift in the resonant frequency observed across all tests almost doubles from approximately 10.4 kHz (Method A) to approximately 20.1 kHz (Method B). The mean shift using Method C is approximately 52.8 kHz and thus approximately five times larger than that observed using Method A. This suggests a substantial improvement in the ability of the system to sample and detect aerosolised particulate matter is achieved with the sampling channel comprising only the microfan and especially with the sampling channel comprising both the microfan and the thermophoretic deposition device. Based on the results in Table 5.1, the improvement in the FBAR sensitivity is especially significant at the lower particle concentrations tested. At 1000 µg/m³ the frequency shift increases by approximately eleven-fold between Method A and Method C.

To assess the difference in the repeatability of the frequency readings, Table 5.2 shows the spread of the results in Fig. 5.26 as the difference between the maximum and minimum frequency shift observed at each particle concentration.

Table 5.2. Mean Frequency Shift Results Spread for the Results in Fig 5.26.

<table>
<thead>
<tr>
<th>Particle Concentration (µg/m³)</th>
<th>Result Spread Method A (kHz) (% of Mean Shift)</th>
<th>Result Spread Method B (kHz) (% of Mean Shift)</th>
<th>Result Spread Method C (kHz) (% of Mean Shift)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>5.9 (111)</td>
<td>1.7 (57)</td>
<td>6.1 (22)</td>
</tr>
<tr>
<td>1000</td>
<td>2.0 (63)</td>
<td>6.2 (42)</td>
<td>8.3 (23)</td>
</tr>
<tr>
<td>2000</td>
<td>7.1 (113)</td>
<td>3.9 (15)</td>
<td>4.3 (8)</td>
</tr>
<tr>
<td>3000</td>
<td>6.2 (39)</td>
<td>2.6 (13)</td>
<td>4.9 (7)</td>
</tr>
<tr>
<td>4000</td>
<td>6.5 (30)</td>
<td>10.3 (27)</td>
<td>6.1 (8)</td>
</tr>
<tr>
<td>Mean Freq. Spread (kHz)</td>
<td>5.5</td>
<td>4.9</td>
<td>5.9</td>
</tr>
<tr>
<td>Mean Spread (% of Mean Shift)</td>
<td>71</td>
<td>31</td>
<td>14</td>
</tr>
</tbody>
</table>
As can be seen from Table 5.2, when comparing the results for Method A, B, and C, the absolute mean spread in the measured resonant frequency shift in Hz only varies by approximately 20 % between the different methods. This suggests that the repeatability of the particle sampling did not vary much between these tests. One possible reason for this could be the high particle concentrations used in these experiments, which would likely have led to some degree of coverage of the sensor with particles even when relying only on random sampling as was done using method A. According to Table 5.1, the increase in frequency shift achieved by using Method C decreases compared to Method A, as the sensor reaches its saturation point (the particle sensitivity is only increased by a factor of three at 4000 µg/m³). Additionally, the tests were repeated in stable conditions, with a precise control of the particle feed into the chamber. This would have also led to somewhat repeatable patterns in particle settling and this would support the theory of relatively repeatable random particle coverage achieved with each method. In real-world testing the results for each method might look more different from one another.

A stronger indicator for the reliability of the particle measurements is perhaps the relative mean spread in the results. This is because, for example, an absolute frequency shift variability of 5 kHz would make the different pollution levels more difficult to distinguish when the maximum frequency shift measured across a specific range of particulate matter levels is 20 kHz, as opposed to 70 kHz. In fact, Table 5.2 shows a strong decrease in the mean variability of the results as a percentage of the observed frequency shifts. Method A experienced mean variations of 71 % on average, while Method B only had 34 % and Method C only 14 % mean variation between the repeat results for each particle concentration tested here. In Fig. 5.26 it can be seen that this means less overlap on the frequency axis between the data points for each particle concentration level, especially at the lower ones. A recorded frequency shift may hence be attributed to a specific level of particle concentration with greater accuracy. Because of this, it can be said that adding the thermophoretic sampling channel with the microfan did help to provide more reliable estimates of the particle concentration.

The need for sensor regeneration at regular intervals is a disadvantage of particle sensors based on FBAR technology, especially when comparing them to sensors which do not require particle capture, but can measure aerosolised particles, e.g. optical particle counters (OPCs) [4]. This is the case especially at very high concentrations of aerosolised particulate matter [4]. Optical
sensors sample the particles in an airstream, the laser then counts the particles, and the airstream is ejected from the sensor mostly unaffected [4]. This approach does not require sensor cleaning following each use [4], although some OPCs do undergo periodic cleaning cycles. In contrast, without outside influence, the particles that bond onto the surface of an FBAR sensor would typically remain there [4].

Some reversibility of the particle sampling was observed during the tests in section 5.2.5 [4]. In Fig. 5.21 and in Fig. 5.22 it can be observed that for the lower particle concentrations the device begins to return to its original base-line of resonant frequency once feed of particles was disabled and the level of aerosolised particles inside the chamber begins to drop through particles settling at its bottom or being passed through the outlet [4]. This can be explained by the continued operation of the sampling fan during this phase of the test, which hence blew some of the particles off the FBAR’s surface [4]. This in turn caused an increase in its resonant frequency as the sensor’s mass loading decreased, and hence made the process of particle sensing reversible [4]. As it was mentioned in section 2.3.2 compressed air is commonly used to clean particulates off device surfaces and the microfan is essentially an embodiment of this principle. The optical sensor for particulate matter SPS30 by Sensirion is one commercial example that uses this cleaning mechanism [4]. It has a sensor cleaning mode realised as a high-speed setting of the sampling fan [4]. The tests in according to Method B and Method C in section 5.2.5 show a loss of the effectiveness of the fan-based sensor cleaning at the higher end of the tested particle concentrations [4]. This is particularly apparent in Fig. 5.22, where there is no visible return of the recorded frequency values to the baseline from the beginning of the tests at 3000 µg/m³ and 4000 µg/m³. This is clearly visible in Fig. 5.27, where the inside of the sampling channel is shown in different states [4].
Figure 5.27. Frequency Sampling Channel (a) Before Test (b) After 1000 µg/m³ Test (c) After 4000 µg/m³ Test [4].

Figure 5.27a shows the clean microchannel [4]. After a test with a particle feed rate of 19 µgm⁻³s⁻¹ (1000 µg/m³ test) some particles remain inside the sampling channel, but it is mostly clean, as is shown in Fig. 5.27b [4]. Figure 5.27c shows a significantly less clean sampling channel after testing the device with a particle feed rate of 94 µgm⁻³s⁻¹ (4000 µg/m³ test) [4]. It is obvious that the resonant frequency of the FBAR should have remained lower in the case of Fig. 5.27c compared to that of Fig. 5.27b [4]. In the former case the microfan clearly was not capable of cleaning the FBAR and Fig. 5.27c illustrates that the observed cleaning effect does not work at higher particle concentrations, most likely because of a clogging of the microfan’s outlet into the microchannel [4]. Nevertheless, the possibility of FBAR cleaning using a microfan seems to be feasible at lower concentrations [4]. The use of a stronger microfan might also enable the reversibility of the particle sampling mechanism for higher particle concentrations. For air quality monitoring this might not be required, because the particle concentrations encountered in such an application is likely to be much lower than the ones tested here and the provided microfan could suffice for sensor regeneration [4]. The use of the existing fan as the cleaning mechanism for the FBAR sensor could further reduce the cost of operation of the sensing device [4].

5.3 Chapter 5 Conclusions
This chapter has presented the improvement in the performance of a FBAR particle sensor achieved at different PM concentration levels by the addition of a particle sampling channel comprising microfan for air sampling and a microheater for thermophoretic particle deposition. The first part
of the chapter described the design and construction of a rig for the testing of particle sensors. It comprised an aerosol generator from a controlled injection of particles into a steady airstream that is fed into a glass chamber to create repeatable variable PM levels. Commercial reference sensors were used to record the particle concentration in the air inside the chamber, as well as the temperature and humidity monitoring. The second part of the chapter describes FBAR sensors supplied by SilTerra used to demonstrate their applicability in PM monitoring as well as the functionality of the thermophoretic sampling channel comprising microhotplates provided by the Materials Center Leoben. The FBAR sensor’s responsiveness to mass loading with Arizona dust particles was initially confirmed without the use of the sampling channel. As expected, the resonant frequency of the FBAR was found to drop increasingly in response to an increasing concentration of particles inside the test chamber. The addition of the particle sampling channel increased the mean frequency shift observed during the tests by a factor of approximately five and was able to increase the sensitivity of the system to aerosolised particles to approximately double overall. Furthermore, the mean relative spread between repeat tests at each particle concentration tested was found to have been reduced from 71% to 14%, which results in a sensor that can be used to discriminate between different levels of aerosolised particulate matter with greater accuracy. The suitability of the sensing system for air quality measurement was hence improved substantially. Additionally, the thermophoretic sampling channel can easily be manufactured at a large scale and thus improve the FBAR particle sensor without the need to adjust the sensor itself. Furthermore, the microfan was found to have strong potential for FBAR sensor cleaning applications, especially at low particle concentrations, as the tests demonstrated.
Chapter 4 presented simulation results demonstrating the functionality of a novel, CMOS compatible solidly mounted resonator (CMOS SMR) with an integrated microheater for temperature modulation. As previously mentioned, this chapter will present the results of experimental tests of the CMOS SMR as a particle sensor. Furthermore, a novel mechanism to increase the frequency sensitivity of the SMR sensors to particle deposition through modulation of the sensor temperature is presented and tested experimentally [18]. The addition of the thermophoretic sampling channel described in chapter 5 to the thermally modulated CMOS SMR to further increase the system’s performance is also evaluated [18]. Section 6.1 begins by describing the changes made to the sensing system configuration to include the thermal modulation. The work in this chapter was published in *IEEE Transactions on Instrumentation and Measurement* in [18].

6.1 Drive and Readout Circuitry

The results of the simulations presented in section 4.2.3 demonstrated that particle deposition may alter the thermal time constant of the CMOS SMR. In practice this change to the temperature gradient should also influence the transient change in the CMOS SMR’s resonant frequency [18]. To assess whether this can improve the CMOS SMR’s sensitivity to particle deposition, the FBAR sensing system from chapter 5 was adapted. Circuitry to drive the integrated microheater to test the device with temperature modulation was included [18]. The updated particle sensing system used the same Pierce oscillator circuit for frequency measurement of the CMOS SMR that was used for the SilTerra FBAR in chapter 5 [18]. The measurement of the Pierce oscillator’s output frequency was also done by using a differential configuration, comprising a sensing and a reference SMR. Circuitry was added to the SMR drive PCB, to control the current through the integrated microheater of the CMOS SMR [18]. A system schematic of the updated drive circuitry is shown in Fig. 6.1.
Fig. 6.1. Differential Configuration with Gain/Phase Detector and Microheater Control.

The circuitry used to control the integrated microheater was the same as that used to control the microhotplates, as was shown in Fig. 5.14. However, instead of a potentiometer, fixed value resistors were used for current control in the source follower. Like in chapter 5, a Mini-Circuits RSM-30+ mixer was used to output a signal with the frequency difference between the sensing and the reference oscillators [18]. Using the mixer circuit meant this frequency difference was substantially lower than the individual oscillators’ frequency and could be measured with a Teensy 3.6 microcontroller after passing through an ADCMP600 comparator. The comparator turned the signal into a square wave with a signal strength at the correct logic level for measurement. Additionally, an AD8302 gain and phase detector was added to the system to measure any changes in the differential signal strength between the sensing and the reference SMR [18].

The CMOS SMR was wire bonded onto a PCB, which also carried the circuitry for the two Pierce oscillators needed to drive the resonators, as well as connections to the microheaters (Oscillator PCB), by Mr Frank Courtney at the University of Warwick. Again, the sensing device was mounted on the upside of the PCB and the reference device on the downside [18]. This way particles were hindered from landing on the surface of the reference sensor. A photograph of the oscillator PCB with the sensing SMR visible is shown in Fig. 6.2 [18].
Thermally conductive silicone paste was used to affix the SMRs reliably to withstand a large range of temperatures [18]. The Oscillator PCB was mounted onto another PCB (Drive Circuit PCB) with Micro-miniature coaxial (MMCX) connectors [18]. The Drive Circuit PCB carried the current source for the microhotplates and the frequency measurement circuitry [18]. A Teensy 3.6 microcontroller for frequency measurement was also connected to it from the underside [18]. The Teensy also measured the output voltage of the gain and phase detector via its on-chip analogue-to-digital converters and provided a regulated 3.3 V power supply [18]. The LabView interface from chapter 5 was used to record the measurement data from the Teensy with a sampling
frequency of 3 Hz, as was shown in Fig. 5.11. The temperature and switching frequency of the current sources were also controlled through LabView via the Teensy, too [18]. As in chapter 5, the particle tests were initially conducted allowing random settling of particles on the sensor surface, to compare and confirm whether the fan-driven, thermophoretic microchannel would be able to improve the particle sensing results for the novel CMOS SMR, too [18]. The complete system setup with the thermophoretic channel is shown from viewed from the back in Fig. 6.3 and from the top in Fig. 6.4 [18].

Fig. 6.3. Setup of Particle Sensor with Microchannel (Back-View) [18].
As shown in Fig. 6.3, the MCL microhotplates described in chapter 5 were glued and wire-bonded onto a PCB (Microhotplate PCB) that was plugged into the Drive Circuit PCB using standard pin headers with a spacing of 2.54 mm [18]. The Microhotplate PCB and the Oscillator PCB formed the top and bottom walls of the microchannel, respectively [18]. A new microchannel was 3-D printed to slide onto the Oscillator PCB and the microhotplate PCB and provided the side walls for the channel, as well as its inlet and a holder for the microfan. The 3-D printed microchannel was redesigned to fit the adjusted Oscillator PCB with the assistance of Mr Frank Courtney at the University of Warwick [18]. The channel, the pin headers and the MMCX connectors ensured a tight fit of the system as a single sensing unit [18].
An AC to DC rectifier was used to power the setup with a 12 V supply from the 220 V mains [18]. When the MCL microhotplates were heated to approximately 673 K (4 mA current supply) and the microheaters of the SMR are powered with a duty cycle of 50 % and heated to approximately 323 K with a pulsed current supply switching between 0 mA and 33 mA, the complete system had a power draw of approximately 550 mW [18].

6.2 Experimental Procedure
To establish whether temperature modulation can be used to increase the sensitivity of the CMOS SMR to particle concentration, the device was initially tested without using the integrated microheater and relying only on random particle settling on the sensing electrode [18]. The sensor was tested in the particle test rig described in section 5.1, as shown in the picture in Fig. 5.10 [18]. In the same manner as in section 5.2, a stream of compressed air was fed into the chamber throughout each test keeping the pressure, humidity, and temperature inside the chamber consistent [18]. Particles were aerosolised into the airstream by the SAG410/L with concentrations depending on the feeding belt setting [18]. In this set of experiments 2, 5, 10, 15 % of the potential maximum belt speed were used. These corresponded to settings of 0.1, 0.25, 0.5 and 0.75 V [18]. Again, three tests were repeated for each aerosol generator setting [18]. Once enabled the SAG419/L injected particles into the airstream for five minutes, after the chamber pressure, humidity and temperature had stabilised [18]. As with the tests described in section 5, the maximum particle concentration measured with the Alphasense OPC-N2 for each of the tested settings was approximately 500, 1000, 2000 and 3000 µg/m$^3$ [18]. After each test the chamber was cleaned to remove the settled particles [18]. Following this, another set of tests, this time with thermal modulation employed with a maximum sensor temperature of approximately 323 K, relying on random particle settling was carried out [18]. For the proof-of-concept tests it was decided that the sensor should be tested at a temperature that was elevated, but not significantly higher than the ambient temperature. This was to ensure that the sensor would withstand all the repeat tests without suffering breakage or excessive permanent deformation from the repeated thermal expansion of the device [18]. A photograph of the SMR surface was taken with a 5 MP Leica ICC50 W camera mounted onto a Leica DM750M microscope with 20× zoom after each repeat test [18]. The mass of particles on the SMR was estimated by cropping the images down to the top electrode and binarisation of the cropped image using MATLAB®. The coverage fraction could be calculated by dividing the number of black pixels by the total number of pixels in the resultant image. This value could be used to calculate the area on the top electrode that was covered in particles and multiplication of
this by the mean diameter (6 µm), and its standard deviation (4 µm), and the density (2500 kg/m³) of the Arizona dust resulted in an approximation of the particle mass [18]. The sensing system was cleaned using compressed air to regenerate the CMOS SMR between tests [18]. The microchannel including the MCL microhotplates was then added to the system and another set of three tests for each concentration was run with temperature modulation, to assess if the performance of the device could be enhanced further.

6.3 Experimental Results for CMOS SMR Particle Sensor

6.3.1 CMOS SMR without Temperature Modulation

As specified above, the sensor relied on the random settling of particles in the first set of tests. An image of the cropped top electrode of the CMOS SMR, as well as the binarized version of the same, at each of the four particle concentrations, is shown in Fig. 6.5 [18].

![Image of the cropped top electrode of the CMOS SMR](image)

**Fig. 6.5.** Original (Top) and Binarised (Bottom) Image (Leica ICC50 W) of Top Electrode After Tests with Increasing Particle Feed Rate from (a) 9.5 (b) 19 (c) 43 (d) 66 µg/m³’s [18].

Maximum concentrations of PM10 particles of approximately 500, 1000, 2000 and 3000 µg/m³ were measured by the Alphasense OPC-N2 commercial particle sensor [18]. From Fig. 6.5 it can clearly be seen that, as the particle concentration increased, the number of settled particles settled on SMR’s top electrode increased significantly, too [18]. Slight variations in the manual
sensor positioning under the camera that was required after each test, caused some slight variations in the focusing and lighting conditions of the images at the top of Fig. 6.5. However, this did not influence the binarised images, see Fig. 16 [18].

Figure 6.6 shows the normalised frequency of the output signal of the mixer circuit that was sampled over time with the Teensy 3.6 for a typical test run without temperature modulation for each particle feed setting [18].

![Fig. 6.6. Normalised, Filtered Mixer Output Signal without Temperature Modulation with Vertical Lines Indicating Duration Time of Particle Injection into the Chamber [18].](image)

The black, vertical lines in Fig. 6.6, at 30 and 35 minutes, indicate the times at which the particle feed was enabled and disabled. During the tests there was no visible signal drift, apart from some variations in the resonant frequency caused by random noise. To filter this from the signal, the results in Fig. 6.6 were processed with a 255-point moving average filter [18]. After the particle feed was disabled, it took approximately 30 minutes for the particle concentration to approach the level it was at before each test as measured with the Alphasense OPC-N2 [18]. During this time the particles either left the chamber in the outlet airstream or they settled down at the bottom of the
chamber and on the sensor [18]. The latter caused an additional, slow drop in the CMOS SMR’s resonant frequency that occurred following the faster drop observed whenever the particle feed was enabled in this set of experiments, see Fig. 6.6 [18]. As can be seen in Fig. 6.6, the sensor experienced shifts in resonant frequency (calculated as the difference between the average frequency measurement before and after the particle injection into the airstream was enabled; rounded to the nearest 1 kHz) of 10 kHz, 17 kHz, 26 kHz, and 61 kHz without temperature modulation, as the particle concentration inside the chamber increased from 500 µg/m\(^3\) to 3000 µg/m\(^3\) [18]. The Teensy 3.6 was set to measure the frequency to the nearest 1 kHz, giving the frequency measurements an uncertainty of ±1 kHz.

Figure 6.7 shows the internal relative humidity and Fig. 6.8 the internal temperature of the test chamber during a 3000 µg/m\(^3\) test [18].

![Graph showing variation of test chamber humidity and frequency during experiment](image.png)

**Fig. 6.7.** Variation of Test Chamber Humidity During Experiment [18].
Fig. 6.8. *Variation of Test Chamber Temperature During Experiment* [18].

There is a variation in the humidity inside the test chamber within approximately 0.5 %, but there is no visible correlation between this and the measured frequency [18]. Similarly, the temperature inside the test chamber varies within less than 0.1 K and there is no observable correlation with the frequency measurements [18]. Hence it can be concluded that the observed frequency shift is caused by particle deposition.

Additionally, without temperature modulation there was no observable change in the signal strength, see the measurement in the gain difference between the sensing and the reference oscillator circuits’ output signals shown Fig. 6.9
Fig. 6.9. *Normalised, Filtered Difference in Signal Gain without Temperature Modulation.*
6.3.2 CMOS SMR with Temperature Modulation

Figure 6.10 shows the raw data for the same result as Fig. 6.7, but for a set of tests with a thermally modulated sensing SMR [18]. Figure 6.11 shows the data from Fig. 6.10 filtered with a 255-point moving average filter.

Fig. 6.10. Normalised Mixer Output Signal with Temperature Modulation with Vertical Lines Indicating Duration Time of Particle Injection into the Chamber.
As can be seen in Fig. 6.11, the frequency shift measured during these tests increased with respect to the tests without temperature modulation to approximately 17 kHz, 47 kHz, 126 kHz, and 135 kHz [18]. Again these have a measurement uncertainty of ±1 kHz. To better compare the results from the tests with and without temperature modulation, the frequency shifts measured during the repeated tests are plotted against the mass of the particles on the sensor surface, estimated from the binary images as described in section 6.2, with error bars representing the possible effect of the standard deviation in particle diameter in the sample particle mix, in Fig. 6.12 [18].
Figure 6.12 shows that an increase in the deposited particle mass on the top electrode corresponds to a resultant increase in the measured shift in the resonant frequency of the device [18]. Hence the frequency shift and the deposited particle mass are proportional to each other. This is also indicated by the positive gradient of both lines of best fit [18]. It can be concluded that the functionality of the novel CMOS SMR as a sensor for particle measurement looks very promising [18]. The error bars in Fig. 6.12 correspond to the particle mass calculated for the upper and lower standard deviation of the particle diameter (approximately 4 µm). For the data in Fig. 6.12, the gradient of the line of best fit without temperature modulation is 40 Hz/ng, see (6.1) [18]. The gradient increases by a factor of almost five when the sensing SMR is modulated. Equation (6.2) gives a gradient for the respective line of best fit of 190 Hz/ng, see (6.2), which indicates a considerable increase in the particle sensitivity of the sensor when thermal modulation is applied [18]. Possible reasons for this increase include a greater thermal loss caused by the particles, as
well as thermal expansion of the particles on the sensor surface and a reduction in the sensor’s quality factor caused by the heating, which might have led to a greater susceptibility of the acoustic wave to particle deposition on the sensor surface. However, further tests and theoretical analysis of the sensor, for example under different temperature conditions and with more targeted mass deposition are required to establish a more precise cause of the observed sensitivity increase. In both (6.1) and (6.2) $\Delta f$ represents the measured shift in resonant frequency, while $m$ is given by the particle mass deposited on the top electrode [18].

$$\Delta f_{\text{non-modulated}} = 40m - 4000$$  \hspace{1cm} (6.1)

$$\Delta f_{\text{modulated}} = 190m - 7500$$  \hspace{1cm} (6.2)

Equation (6.3) and (6.4) are versions of (6.1) and (6.2) with variables replacing the gradients ($a_n$ and $a_m$ for the non-modulated and the modulated sensor, respectively) and the $y$-intercepts ($b_n$ and $b_m$ for the non-modulated and the modulated sensor, respectively).

$$\Delta f_{\text{non-modulated}} = a_n m - b_n$$  \hspace{1cm} (6.3)

$$\Delta f_{\text{modulated}} = a_m m - b_m$$  \hspace{1cm} (6.4)

The values for $a_n, a_m, b_n$ and $b_m$ calculated from the mean particle size and its standard deviation are shown in Table 6.1, when input into (6.3) and (6.4) they give the equations for the lines of best fit for the data points (crosses) in Fig. 6.12, as well as each end of the error bars.

**Table 6.1. Values for $a_n, a_m, b_n$ and $b_m$.**

<table>
<thead>
<tr>
<th>2 µm Particle Height</th>
<th>Mean Particle Height</th>
<th>10 µm Particle Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>$a_n$</td>
<td>110</td>
<td>40</td>
</tr>
<tr>
<td>$b_n$</td>
<td>4100</td>
<td>4100</td>
</tr>
<tr>
<td>$a_m$</td>
<td>500</td>
<td>190</td>
</tr>
<tr>
<td>$b_m$</td>
<td>7500</td>
<td>7500</td>
</tr>
</tbody>
</table>

It can be seen from this that there is considerable uncertainty in the estimates for the increase in particle sensitivity of the sensor (assumed to be $a_n$ and $a_m$) achieved by adding thermal modulation. For example, the minimum value estimated for the thermally modulated device is less than 10% higher than the maximum value estimated for the unmodulated sensor. Future test with
a more precise size-control of the sample particles could potentially reduce this uncertainty. Furthermore, a detection limit of 100 ng of particles when the sensor is not thermally modulated can be approximated from the x-intercept of the lines of best fit in Fig. 6.12 [18]. This detection limit decreases by approximately half to 50 ng of particles with temperature modulation [18]. The thermal modulation has thus resulted in increased sensitivity of the CMOS SMR to particles [18]. Additionally, the difference in the signal strength between the sensing and the reference oscillator circuit’s output signals is shown below in Fig. 6.13, when the sensing SMR is thermally modulated.

![Graph showing normalised, filtered difference in signal gain with temperature modulation](image)

**Fig. 6.13. Normalised, Filtered Difference in Signal Gain with Temperature Modulation.**

Unlike for the gain measurements made during the tests with no temperature modulation, see Fig. 6.9, a reduction in in the normalised signal strength of the sensing oscillator after particle deposition is clearly visible in Fig. 6.13. The signal strength of the sensing oscillator decreased with respect to the reference oscillator by 0 dB, 0.1 dB, 0.15 dB and 0.2 dB corresponding to the 500, 1000, 2000 and 3000 µg/m³ maximum particle concentrations, respectively [18].
6.3.3 CMOS SMR with Thermophoretic Sampling Channel

To see if the temperature modulation could be used together with the fan-driven sampling channel and the thermophoretic particle deposition system the tests were then repeated with the complete setup as shown in Fig. 6.4 [18]. The frequency shift from the repeat tests for all three sets of measurements are shown plotted against the maximum article concentrations as measured with the Alphasense OPC-N2 in Fig. 6.14 [18].

![Graph](image)

**Fig. 6.14. Resonant Frequency Shift vs Particle Concentration in Chamber** [18].

The lines fitted to the data were based empirically on the Langmuir isotherm model, because a saturation effect like the one observed in Fig. 5.26 is also visible in Fig. 6.14 [18]. It’s coefficients for calculation of the saturation constant are given by $R$ and $N$, where $\Delta f_S$ is the frequency shift corresponding to a particle concentration of $C$.

$$\Delta f_S = \frac{R \times N \times C}{1 + N \times C}$$  \hspace{1cm} (6.4)

The equations for the fits for the three sets of tests are given by (6.5), (6.6) and (6.7) below [18].
\[ \Delta f_{\text{Non-modulated}} = \frac{9.1 \times 10^4 \times 2.0 \times 10^{-4} \times C}{1 + 2.0 \times 10^{-4} \times C} \]  

(6.5)

\[ \Delta f_{\text{Modulated}} = \frac{5.0 \times 10^5 \times 1.3 \times 10^{-4} \times C}{1 + 1.3 \times 10^{-4} \times C} \]  

(6.6)

\[ \Delta f_{\text{Channel}} = \frac{6.7 \times 10^4 \times 9.8 \times 10^{-4} \times C}{1 + 9.8 \times 10^{-4} \times C} \]  

(6.7)

The error in the coefficients \( R \) and \( N \), based on the standard deviation of the frequency measurements, is displayed in Table 6.2. the subscripts \( n \), \( m \) and \( c \) stand for tests without modulation, with modulation and with the channel, respectively.

**Table 6.2. Standard Deviation Curve Fit Coefficients for Fig. 6.14.**

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Coefficient Value for Standard Deviation Fit</th>
</tr>
</thead>
<tbody>
<tr>
<td>( R_n )</td>
<td>( 1.6 \times 10^6 )</td>
</tr>
<tr>
<td>( N_n )</td>
<td>( 3.9 \times 10^{-6} )</td>
</tr>
<tr>
<td>( R_m )</td>
<td>( 2.8 \times 10^4 )</td>
</tr>
<tr>
<td>( N_m )</td>
<td>( 2.1 \times 10^{-3} )</td>
</tr>
<tr>
<td>( R_c )</td>
<td>( 1.8 \times 10^6 )</td>
</tr>
<tr>
<td>( N_c )</td>
<td>( 3.7 \times 10^{-6} )</td>
</tr>
</tbody>
</table>

These values show that the standard deviation of each coefficient and thus the uncertainty of the fit is largest for the tests with the thermally modulated SMR, which is mostly likely attributable to the significantly larger shifts but can also have been caused by the random particle positions, for example. Section 6.4 will discuss the improvement in sensor reliability achieved by adding thermal modulation in more detail.

It can be seen from Fig. 6.14 that the results from the tests using the channel with temperature modulation showed an improved response of the sensor compared to the results that were recorded without the temperature modulation and without the channel [18]. For example, the maximum frequency shift on the fitted curve was approximately 50 kHz with the microchannel and the modulation, while it was approximately 35 kHz without either [18]. A reduction in the efficiency of the additional particle sampling and deposition mechanism at higher particle concentrations is
clearly visible in Fig. 6.14 [18]. The increase in the spread of frequency shifts (20 kHz at 2000 µg/m³ compared to ~40 kHz at 3000 µg/m³), as well as the reduction in their magnitude (50 to 70 kHz at 2000 µg/m³ compared to 20 to 60 kHz at 3000 µg/m³) at the higher end of the tested concentrations indicated this [18].

6.4 Improvement in Sensor Performance

There was a marked improvement in the sensitivity of the CMOS SMR to particle deposition when using the temperature modulation mechanism [18]. However, the measured shifts in the resonant frequency of the SMR were lower with the microchannel than they were without (up to 70 kHz with the channel compared to up to 160 kHz using temperature modulation only) [18]. To compare the results, Table 6.3 lists the mean of shifts in the resonant frequency of the sensor recorded at each particle concentration that were plotted in Fig. 6.14, as well as their standard deviation.

Table 6.3. Mean Frequency Shift Results in Fig. 6.14 (Standard Deviation in Brackets).

<table>
<thead>
<tr>
<th>Particle Concentration (µg/m³)</th>
<th>Frequency Shift No Modulation (kHz)</th>
<th>Frequency Shift Modulation (kHz)</th>
<th>Frequency Shift with Channel (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>10 (2)</td>
<td>10 (4)</td>
<td>20 (2)</td>
</tr>
<tr>
<td>1000</td>
<td>13 (3)</td>
<td>50 (32)</td>
<td>30 (4)</td>
</tr>
<tr>
<td>2000</td>
<td>27 (13)</td>
<td>132 (26)</td>
<td>62 (10)</td>
</tr>
<tr>
<td>3000</td>
<td>35 (23)</td>
<td>128 (16)</td>
<td>41 (21)</td>
</tr>
<tr>
<td>Mean Shift (kHz)</td>
<td>21</td>
<td>80</td>
<td>38</td>
</tr>
</tbody>
</table>

The mean frequency shift, and hence the mean frequency sensitivity to particle deposition, measured with the temperature modulation was approximately 80 kHz, while it was only approximately 21 kHz without modulation. This was an improvement by a factor of approximately four [18]. There is no improvement in the mean shift at 500 µg/m³. However, based on the mass estimate in Fig. 6.12, the actual mass of particles that the frequency shift value of 10 kHz with modulation corresponds to was approximately 100 ng of particles, as opposed to the approximately 450 ng of particles that caused the same level of mean frequency shift without temperature modulation. Discrepancies in particle masses were mainly due to the random settling of the particles, which reduces the repeatability of the results, as described above [18]. It can be seen in Fig. 6.12 that this difference in particle mass when comparing the tests with the modulated and unmodulated sensor was relatively less at the higher concentration, because of the larger mass of particles deposited. This led to a more substantial increase in the mean frequency shift based on
the level of aerosolised particles. Consequently, the mass sensitivity approximated from the lines of best fit in Fig. 6.12 is increased by a factor of five rather than four, from 40 Hz/ng to 190 Hz/ng, as they use a more direct estimate of what the sensor is detecting compared to Fig. 6.14 [18]. This was also accompanied by a reduction in the estimated detection limit of the sensor by approximately 50 %, from 100 ng to 50 ng [18].

Adding the microchannel to the thermally modulated CMOS SMR led to a decrease in the mean frequency shift measured. At 38 kHz it was approximately half of that observed without the channel. The microchannel does not seem to have improved the sampling efficiency of the particles in this case, since the mean frequency shifts measured at each concentration were not increased, except for the tests that were run at 500 µg/m³ particle concentration. Here the mean frequency shift did increase from 10 kHz to 20 kHz. This was expected from the saturation of the particle sensing system at higher particle concentrations that was also observed using the SilTerra FBAR in chapter 5. Additionally, especially at lower particle concentrations, the repeatability of the results was improved using the microchannel [18]. The spread in the measurements at each concentration illustrates this in Table 6.4.

Table 6.4. Mean Frequency Shift Results Spread for the Results in Fig. 6.14.

<table>
<thead>
<tr>
<th>Particle Concentration (µg/m³)</th>
<th>Result Spread No Modulation (kHz) (% of Mean Shift)</th>
<th>Result Spread Modulation (kHz) (% of Mean Shift)</th>
<th>Result Spread with Channel (kHz) (% of Mean Shift)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>3 (33)</td>
<td>6 (60)</td>
<td>4 (20)</td>
</tr>
<tr>
<td>1000</td>
<td>5 (38)</td>
<td>50 (100)</td>
<td>7 (67)</td>
</tr>
<tr>
<td>2000</td>
<td>25 (93)</td>
<td>50 (39)</td>
<td>20 (32)</td>
</tr>
<tr>
<td>3000</td>
<td>44 (126)</td>
<td>30 (23)</td>
<td>40 (98)</td>
</tr>
<tr>
<td>Mean Freq. Spread (kHz)</td>
<td>20</td>
<td>34</td>
<td>18</td>
</tr>
<tr>
<td>Mean Spread (% of Mean Shift)</td>
<td>73</td>
<td>56</td>
<td>54</td>
</tr>
</tbody>
</table>

With the microchannel the measured spread in frequency shifts was comparable to that measured without the channel and without temperature modulation. However, with the microchannel this spread was observed for significantly larger frequency shifts. This also explains the generally larger standard deviation observed in Table 6.4 to some extent, although the main cause of this probably was the randomised particle settling. The relative spread decreased by approximately 26 %. Furthermore, the steeper curve of frequency shifts, as plotted in Fig. 6.14,
does indicate that this system is better placed to discriminate different levels of particulate matter more reliably [18]. This matches the findings from chapter 5.

Without appropriate particle sampling means repeatable measurements of aerosolised particulate matter using a CMOS SMR were difficult to achieve. Even when the overall level of particulate matter was controlled, there was a large variation in the deposited particle masses between repeat tests, see Fig. 6.12. There was an increase in the absolute spread of the frequency shift measurements from a mean of 20 kHz to a mean of 34 kHz when temperature modulation was applied to the CMOS SMR. However, the relative spread as a proportion of the mean frequency shift decreased from 73 % to 56 % during this set of experiments. This was a result of the fivefold increase in the frequency sensitivity of the sensor, which meant that a higher shift could sustain a more significant spread in the frequency results while still being capable of improved discrimination of PM levels.

The relative spread as a proportion of the mean shift in resonant frequency shift measurements was not reduced substantially by adding the microchannel to the thermally modulated CMOS SMR. However, there was a reduction in the absolute spread by approximately half. This indicates an improvement in the particle sampling repeatability of the system, as the thermophoretic microchannel gave the system better control of the positioning and the mass of the particles that were deposited on the SMR’s surface [18]. At the lower particle concentrations this reduction was particularly evident. However, at 3000 µg/m³ there was an increase in the spread of the frequency shifts and a substantial reduction of the measured frequency shifts [18]. The spread of the frequency shifts was approximately 7 kHz with thermal modulation, which the addition of the channel reduced to approximately 4 kHz. At 1000 µg/m³ the channel reduced this value from 50 kHz to 7 kHz, while it reduced it from 50 kHz to 20 kHz at 2000 µg/m³, which led to an overall reduction from 36 kHz down to 10 kHz at these three particle concentrations [18]. Considering that the frequency shifts observed at 500 µg/m³ were higher with the microchannel than they were without suggests that the combination of temperature modulation and the thermophoretic particle sampling channel might be more suited to applications where the particle concentration in the air is lower than it was in these tests, which is very promising for practical applications [18].

6.5 Discussion

The main aim of this chapter was to demonstrate the potential of using SMR devices, fabricated using a process that is suitable for mass-fabrication, for air quality measurement by increasing their
sensitivity to particulate matter deposition through temperature modulation [18]. The experimental findings presented here built on the simulation results presented in Chapter 4 and showed promising results [18].

The particle sensing simulations in section 4.1.3 were performed prior to fabrication of the SMR sensors [18]. As stated previously, there was a discrepancy of approximately 10 % in the resonant frequency calculated in the simulation compared to the one the sensors were found to have practically, which was mainly attributed to differences in the properties of the materials used and variations in the layer thicknesses of the CMOS Bragg reflector of up to 5 % [18].

Compared to the simulations, a unit particle mass deposit on the top electrode caused a practically observed frequency shift that was significantly smaller (approximately 1000 Hz/ng compared to 40 Hz/ng) [18]. Likely causes of this overestimation were some of the model’s assumptions. These included perfect surface coupling of the particles to the SMR’s top electrode, as well as the fact that the simulations considered only the active area of the resonator [18]. The simulated device thus also had a smaller mass relative to the particles than a full, fabricated sensor die [18]. Hence the resonant frequency of the device would have been affected less by the deposition of a comparatively small mass [18]. Furthermore, it is possible that the particle mass in Fig. 6.12 is an overestimation of the true particle mass of the sensor, since the calculation assumed a constant particle diameter, when in reality many particles would have been smaller in the z-direction of Fig. 6.5 [18]. In turn, this would have led to an underestimation of the mass sensitivity of the sensor based on the practical results.

As was shown in the characterisation of the CMOS SMR in section 4.2.3, the value of the sensor’s S21 parameter decreased with increasing temperature through increased resonator damping, which also led to a decrease in the device’s Q value. The result in Fig. 6.13 indicates an additional increase in signal damping through particle deposition when the sensor was modulated thermally. A drop in oscillator signal strength of up to 0.2 dB was only detectable by the AD8302 in case of temperature modulation [18]. This was possibly caused by an increase in the heat loss through the device caused by the increase in its thermal mass and thermal time constant through the particle deposition, as it was observed in the simulations [18]. The decrease in the Q factor of the sensor caused by heating could be one explanation for the higher sensitivity to particles.

Each time the power state of the heater was changed, these factors caused the oscillator’s output frequency to change [18]. This led to a greater sensitivity of the frequency to each unit mass
of particles deposited on the CMOS SMR’s top electrode, see Fig. 6.12 [18]. The shift in the resonant frequency readout was proportional to the particle mass and hence consistent with the Sauerbrey equation [18]. Temperature modulation led to an approximately five-fold improvement in the sensitivity of the CMOS SMR particle sensor, from 40 Hz/ng to approximately 190 Hz/ng, while the sensor’s detection limit was improved from approximately 100 ng to 50 ng [18]. It was observed that the effect of the temperature modulation was amplified at larger particle mass. Since a larger mass of particles affects both the resonant frequency and the thermal characteristics of a sensor more than a smaller mass, the steeper gradient of the line of best fit in Fig. 6.12 was expected [18].

High variability in analyte mass on the SMR surface (up to 250 ng) was observed with random settling of particles, which caused a large spread in the frequency measurements [18]. The addition of the microchannel was observed to increase the repeatability and hence the reliability of the particle sensing system at the lower particle concentrations [18]. The controlled particle deposition through thermophoresis thus seems to make this sensor more suitable for the sensing of aerosolised particles, as it did in chapter 5. However, when using the CMOS SMR, the sampling channel did not cause a substantial increase in the effective particle sampling efficiency, seemingly this was due to a saturation of the microchannel at higher particle concentrations [18]. This could have been caused by increased cooling of the microhotplates by the sampling fan. This might be reducing the thermophoretic force that directs particles onto the sensor and more of these might hence leave the channel “undetected” [18]. Regardless, the observed improvement at lower concentration does make this solution very interesting for further investigation, since these concentrations are closer to those encountered when monitoring urban or rural air quality [18].

When comparing the CMOS SMR and the SilTerra FBAR, the absolute frequency shifts observed were larger for the CMOS SMR than they were for the SilTerra FBAR. The CMOS SMR has a larger active area thus a higher frequency sensitivity than the SilTerra FBAR. For example, CMOS SMR experienced a mean frequency shift of 10 kHz at 500 µg/m³ and the SilTerra FBAR displayed approximately half that at 5 kHz, while the mean shifts were 20 kHz and 10 kHz, respectively. However, the relative spread in the measured resonant frequency shift using random particle settling were similar. It was 71 % for the SilTerra FBAR and 73 % for the unmodulated CMOS SMR. The increase in the repeatability of the frequency shift measurements because of the addition of the microchannel was larger for the tests using the SilTerra FBAR than it was for the CMOS
SMR. This is possibly caused by the smaller ratio of sensing area size and mass to particle size and mass of the SilTerra FBAR compared to the CMOS SMR. The increase in repeatability was the most significant for the difference in the relative frequency shift (14 % compared to 54 %). Thus, a trade-off seemingly exists between frequency sensitivity through a larger device size and the repeatability of the sensor readings, which are both important indicators of its reliability.

The CMOS SMR was powered with a 12 V DC power supply in and the whole system had a power consumption of only 0.55 W. This is significantly lower than the power consumption of the OPC-N2, for example, which is 0.9 W [18]. Furthermore, the system could be powered entirely by a Teensy 3.6 with a 3.3 V battery capable of powering its 0.85 W power supply capability, as it is only approximately two-thirds of its power supply maximum [18]. This significantly increases the SMR system’s suitability for personal air quality measurement, which is an important factor in reducing the health impact of particulate matter pollution [18]. The power draw can potentially be reduced significantly, if the sensor design is optimised by using a heater material with a higher resistivity, or by back-etching the sensor to increase its heating speed and the relative effect of particles on its thermal mass [18].

6.6 Chapter 6 Conclusion
In this chapter a novel, CMOS-compatible, low-cost SMR with an integrated microheater that was fabricated in a standard 180 nm CMOS-BAW process was presented [18]. The device was integrated into a complete low-power sensor system with differential sensor drive and read-out circuitry. In the experiments, temperature modulation of the CMOS SMR was observed to lead to a substantial improvement in the sensitivity of the device to surface mass loading with Arizona dust particles [18]. The mass sensitivity of the CMOS SMR was amplified from 40 Hz/ng without to 190 Hz/ng with the modulation. It was observed that at higher particle concentrations thermal modulation of the microbalance sensor led to a relatively stronger increase in particle sensitivity [18]. Following the practical test of the novel temperature modulation mechanism, a microfan and microhotplates for thermophoretic particle deposition were added to the CMOS SMR as a particle sampling mechanism. This was not found to improve the sensitivity of the CMOS SMR to aerosolised particles by increasing the particle sampling efficiency to the same degree as it did for the SilTerra FBAR. However, it was demonstrated that it led to improved reliability and repeatability of measurements of different particulate matter concentrations [18]. This was especially true at the lower end of the tested PM levels, at which the average spread of the frequency
shift data was shown to improve from 36 kHz to 10 kHz, leading to a higher repeatability of the sensing results [18]. The addition of temperature modulation to resonator particle sensors was thus shown to have significant potential to improve the SMR particle sensing performance to make it more applicable to real world applications [18]. Additionally, the temperature modulation was found to enable discrimination of particulate matter levels based on the differential in the SMR Pierce oscillator signal strength [18].
7 Conclusions and Future Work

7.1 Conclusions

Air quality monitoring is an issue of increasing concern because of the negative impact of human exposure to particulate matter pollution on human health and its increasing economic cost. To mitigate this effect regulatory pollution levels are in place in many countries, but the limited availability of pollution data is currently restricting the effect these regulations can have, as well as the ability of individuals to avoid areas with high pollution more effectively. More extensive air quality data based on widespread real-time pollution monitoring are required to improve this. However, much of the particulate matter monitoring that is currently being carried out by official bodies around the world uses expensive, bulky equipment that is often not capable of providing real-time data with sampling intervals smaller than 24 hours and consumes hundreds of watts of power in some cases. Hence the development of particulate matter monitoring equipment that is better and cheaper has long been recognised as a necessity. Microelectromechanical system (MEMS) technology has been identified as an area with strong potential to resolve many of the issues of current state of the art particle sensing equipment. As the most significant obstacle to a more widespread application of particulate matter monitoring equipment is its cost, especially MEMS sensors manufactured in CMOS compatible processes are of increasing interest, since they may be manufactured cheaply in volume.

The main issue with MEMS particle sensors currently is their sensitivity to particles. MEMS sensors exploiting bulk acoustic wave technology with frequency readout mechanisms, such as film bulk acoustic resonators and solidly mounted resonators have substantial potential to overcome this issue, because they can now achieve very high resonant frequencies of several gigahertz. This has led to the development of sensor devices with substantial mass sensitivity, although for practical pollution measurements this must be increased further still. The combination of MEMS particle sensors with external particle sampling devices was identified as one possibility to achieve this through increasing particle sampling efficiency for each measurement without requiring alterations to the sensors themselves, because it increases the signal to noise ratio of the sensor at a given particle concentration.

The main aim of this research was the development of a novel, CMOS-compatible solidly mounted resonator particle sensor, an improvement of its reliability through targeted particle deposition and the increase of its sensitivity to particle deposition through thermal modulation. A
novel solidly mounted resonator with an integrated microheater for temperature control was designed and simulated for verification prior to its fabrication by SilTerra Malaysia using their combined CMOS-BAW process. The functionality of the CMOS SMR was then evaluated in a set of frequency and thermal characterisation experiments, during which it performed approximately as expected. For example, the practical sensor temperature across different microheater currents, as well as its resonant frequency differed from the simulations by approximately 10%. The CMOS SMR had a resonant frequency of approximately 2 GHz and a Q factor of approximately 200. Its thermal time constant was 1.85 s and the device failed at a microheater current of 125 mA with a surface temperature of approximately 603 K. Functional frequency control of a Pierce oscillator circuit’s output signal through modulation of the SMR that was its frequency setting element was demonstrated.

For the experimental testing of different particle sensors, a test rig was built that could controllably generate different levels of particulate matter concentration inside a glass test chamber based on the feeding belt speed of a commercial aerosol generator, with commercial sensors delivering reference particulate matter concentration measurements. FBAR devices supplied by SilTerra Malaysia were tested with and without a thermophoretic particle sampling channel comprising a microfan and a microhotplate array for more targeted particle deposition on the sensor surface inside this setup. This was done to assess whether thermophoresis could effectively improve the particle sampling efficiency, as well as the repeatability of particulate matter measurements of bulk acoustic wave particle sensors. The FBAR device was found to be capable of detecting particulate matter. The particle sampling channel improved the measured sensitivity of this particle sensing system to aerosolised particles by approximately double overall, while it improved the spread of results between repeat tests from 71% to 14%, which corresponded to a substantial increase in sensor accuracy. The use of thermophoretic particle deposition to improve low-cost MEMS particle sensors was hence found to have significant potential for real-world applications.

The final chapter of this thesis presented the results of experimental particle sensing tests of the novel CMOS SMR sensor that was developed as part of this work. The CMOS SMR was combined with a differential sensor drive and frequency read-out circuitry and a complete low-power sensor system was created this way. The practical particle sensing tests of the CMOS SMR were carried out in the assembled particle test rig with and without the application of temperature
modulation to the sensor by means of the integrated microheater. A substantial increase in the mass sensitivity of the sensing system’s output frequency from 40 Hz/ng to 190 Hz/ng and of its detection limit from 100 ng to 50 ng was observed when temperature modulation was applied to the sensor. The effect was observed to be particularly strong at high particle concentrations. Furthermore, the thermally modulated sensor was found to be capable of detecting particles based on its signal strength, while the non-modulated sensor was not. Adding the thermophoretic particle sampling channel to the CMOS SMR was found to not improve the ability of the CMOS SMR to detect aerosolised particles as much as it did for the SilTerra FBAR, but it did improve the reliability of the sensor readings significantly, especially for lower particle concentrations, where it improved the spread of the measured results by two-thirds. It was thus concluded that temperature modulation and thermophoretic particle sampling can both be used to improve bulk acoustic wave particle sensors, which will help to reduce their limit of detection and hence help to bring low-cost particulate matter monitoring equipment based on this technology closer to the requirements for real world air quality control applications.

7.2 Future Work

Future work to build on the findings from this thesis could include tests of the sensing system at particle concentrations closer to those found in most real-world air quality monitoring applications, which would require some alterations to the test rig to enable the reliable creation of significantly lower particle concentrations in the setup. In-situ testing of the particle sensing system is another possible way of carrying out further tests. In future performance of the device could also be improved by gathering a much larger amount of test data to use regression algorithms and machine learning to enable the better prediction of the particle concentration from a signal with significant noise and interference.

Temperature modulation of the novel CMOS SMR with a higher on-state temperature by applying more power to the heater to assess whether this could improve the sensor beyond the improvements seen from the experiments in this research is another example of possible future work.

Additionally, the sensing setup from chapter 6 could be tested with a range of different particle types, to assess the effect of temperature modulation and thermophoresis on different particle materials or sizes. It could also be tested whether control of the microhotplate temperature could be used for a more targeted thermophoretic effect that might allow particle filtering prior to
particle deposition, which could also be implemented using filters or virtual impactors, to use the
device with an array of SMRs to measure particle type distribution directly. Furthermore, the
different SMR designs including the different electrode and Bragg reflector types that were shown
in chapter 4 could be tested for particle sensing.

Another range of future experiments that could be carried out with the novel CMOS SMR
with temperature modulation is its suitability for other sensing applications, such as the monitoring
of targeted volatile organic compounds, which is also related to air quality monitoring, as well as
biosensing, for example. The SMR would be an additional bio(chemical) sensing layer deposited
on top of the devices.

On top of this, there is potential to implement different types of read-out mechanisms using
this sensor. For example, sensor readout based on its signal strength could be explored further, as
mentioned in chapter 6. Readout circuitry based on phased-locked loop topologies could also be
investigated when temperature frequency modulation is used.

Another possible extension of this research is the optimisation of the design of the CMOS
SMR. For this an optimised Bragg reflector, with greater control of its layer design and material
choice could be developed. Furthermore, the microheater could be optimised, especially in terms
of its metallisation material. The sensor could also be back etched to reduce conduction heat losses
and thus its thermal time constant. This would also increase the sensor’s maximum operating
temperature, which might be exploited for sensor cleaning based on thermal decomposition. These
changes could also lead to lower power consumption of the sensing system, although they might
increase the cost of sensor fabrication reduce its CMOS compatibility. A further increase of the
CMOS SMR’s resonant frequency might also lead to increased particle sensitivity, which makes
this interesting to explore, too.

Full integration of the sensor’s drive and readout circuitry using the same CMOS process as
the sensor could also be done to fabricate full system-on-chip particle sensors, which could reduce
size, fabrication cost and power consumption of the complete system considerably.
References


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