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Original citation:
Thomas, Sanju, Villa-López, Farah Helue, Theunis, Jan, Peters, Jan, Cole, Marina and Gardner, Julian. (2016) *Particle sensor using solidly mounted resonators.* IEEE Sensors Journal, 16 (8). pp. 2282-2289

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This article has been accepted for publication in a future issue of this journal, but has not been fully edited. Content may change prior to final publication. Citation information: DOI 10.1109/JSEN.2015.2512303, IEEE Sensors Journal

Particle Sensor using Solidly Mounted Resonators

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***Abstract*—This work describes the development of a novel particle sensing system employing zinc oxide based Solidly Mounted Resonator (SMR) devices for the detection of airborne fine particles (i.e. PM2.5 and PM10). The system operates in a dual configuration in which two SMR devices are driven by Colpitts type oscillators in a differential mode. Particles are detected by the frequency shift caused by the mass of particles present on one resonator with the other acting as a reference channel. Experimental validation of the system was performed inside an environmental chamber using a dust generator with particles of known size and concentration. A sensor sensitivity of 4.6 Hz per µg/m^3 was demonstrated for the SMRs resonating at a frequency of 970 MHz. Our results demonstrate that the SMR based system has the potential to be implemented in CMOS technology as a low-cost, miniature smart particle detector for the real-time monitoring of airborne particles.**

***Index Terms*— Acoustic wave sensor, air quality monitoring, Colpitts oscillator, particle sensor, particulate matter, solidly mounted resonator (SMR).**

I. INTRODUCTION

AIRBORNE particulate matter (PM10 and PM2.5) consist of a mixture of chemical substances that can be found in the air in the form of very small particles. PM10 refers to those particles that have an aerodynamic diameter equal to or smaller than 10 µm whereas PM2.5 are particles with diameters of 2.5 µm or smaller. Particulate Matter (PM) has been associated with adverse effects on human health and the consequent increase in mortality and morbidity rates [1]. Cardiovascular diseases and respiratory problems such as heart failure and reduced lung capacity have been linked to the exposure to airborne particulate pollution [2]. Other health problems related to PM have been reported, such as diabetes, atherosclerosis, and their impact on birth outcomes [3-5].

In order to reduce human exposure to PM and so minimize their health effects, the U.S. Environmental Protection Agency (EPA) and the European Commission (EC) have issued regulations in which threshold and target values of PMs are defined to maintain them within safe exposure limits. Commercially available instruments for particulate matter detection are generally large in size, expensive and difficult to operate [6]. These instruments are based on several methods and techniques. As an example, filter-based gravimetric samplers such as the Partisol™ Sampler (Thermo Scientific) are used in the UK monitoring network. They draw a sample air through a filter trapping certain particles, which needs to be weighted later in the laboratory [7]. Other automated mass measurement instruments for the continuous monitoring of PM are also available such as the tapered element oscillating microbalance (TEOM™) and Beta gauges. Optical methods mainly based on absorption and scattering of light are the most commonly used for particle detection, counting and size measurement. The Thermo Scientific™ 5030 SHARP monitor and the GRIMM 1.107 monitor (GRIMM Technologies, Inc.) are examples of these type of instruments [8]. Personal sampling instruments such as the the DataRAM™ pDR-1500 (Thermo Scientific) are also currently in the market. Optical techniques, however, are complex and costly as they require the integration of several optical components [9].

For these reasons, a low-cost, real-time and portable particle sensing device is desired and different approaches have been recently proposed. Lim *et al.* [10] reported a MEMS particle detector based on the corona discharge principle, whereas a MEMS electrometer was proposed by Jaramillo *et al.* for the counting of aerosol particles [11]. Park *et al.* [12] developed a particle sensor using a paddle-type silicon cantilever and the use of thin-film piezoelectric on silicon resonators has been proposed by Harrington *et al*. [13]. Thermally actuated MEMS resonators were demonstrated for the mass measurement of airborne particles [9, 14] and the use of such structures within aerosol impactors for the size separation of particles have been proposed as well [15, 16]. The collaboration between the Institute of Semiconductor Technology (IHT) and the Fraunhofer-Wilhelm-Klauditz-Institut led to the development of silicon resonant cantilever sensors for the detection of airborne nanoparticles [17, 18] and further work reported the development of portable cantilever-based detectors [19-21].

Acoustic wave based devices have also been used as an alternative approach for particle sensing. These devices use a piezoelectric material in which a mechanical wave is generated

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Manuscript submitted for review December 23, 2015. This work was funded under the European Commission 7th Framework Programme, Project No. 611887, "Multi-Sensor-Platform for Smart Building Management: MSP".

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when an electrical field is applied. The mass loading onto the resonator due to the particles deposited on the sensing area causes a shift on the resonant frequency of the device. Quartz Crystal Microbalances (QCMs) were used in the system proposed by Liang *et al.* for the measurement of particle mass concentration and size distribution [22] and a sensor based on Surface Acoustic Wave Resonators (SAWR) was demonstrated for the detection of fine particles by Thomas *et al*. [23]. The typical operating frequencies of QCMs are in the range of 5 – 30 MHz [24] whereas the resonant frequency of SAW devices is typically between 30 MHz and 1 GHz [25].

Thin Film Bulk Acoustic Wave devices (TFBAW) make use of thin film technology to operate at higher resonant frequencies and therefore higher sensitivities are achieved compared to other devices. TFBAW devices consist of a thin piezoelectric layer sandwiched between two electrodes and fabricated on top of a carrier substrate, typically silicon. The footprint of TFBAW devices is much smaller to that of SAWs and QCMs and unlike SAW devices, TFBAWs are compatible with low-cost silicon technologies making them suitable for monolithic integration.

In TFBAW, acoustic isolation to the substrate must be provided to the resonator structure in order to confine the wave energy and prevent wave dissipation into the substrate. According to the way in which this is achieved, two different types of TFBAW devices can be differentiated namely, Film Bulk Acoustic Resonators (FBAR) and Solidly Mounted Resonators (SMR).

The use of an FBAR device as the mass sensitive element for the development of a portable particulate matter monitor was proposed by researchers at the University of California, Berkley [6, 26].

In this work, we present the development of a low-cost, highly sensitive particle sensing unit employing zinc oxide based Solidly Mounted Resonators working in a dual configuration and driven by Colpitts type oscillators.

II. DESCRIPTION OF THE SYSTEM

A. Overall Outline

The overall structure of the developed particle sensor system is shown in Fig. 1. The system operates in a dual mode configuration for the suppression of common mode interferences [27], such as temperature, humidity or pressure effects. SMR devices resonating at ~970 MHz are driven by a Colpitts type oscillator, one device is working as the reference channel whereas the second one is acting as the sensing device. The output signal of the oscillators is sent to an interface board that includes an RF mixer, a low pass filter and a comparator. The high frequency signals of the oscillators are mixed and filtered obtaining a differential frequency output easier to measure at high resolution.

The differential signal is ported to a microcontroller for the measurement of the frequency and data are logged to a PC via USB serial communication using National Instruments LabVIEW virtual instrumentation. The assembled particle sensing unit is shown in Fig. 2. Dies containing a total of four SMRs were used but only one of the devices was connected. The SMRs were wire-bonded onto an LCC package and interfaced to the oscillator boards. The reference SMR was covered with a 3-D printed cap. The cap consisted of a thin layer (0.15mm thick) that completely covered the sensor preventing particles to fall onto its surface but also allowing the SMR to be exposed to the ambient conditions, i.e. temperature and humidity. In this way these common mode interferences can be suppressed by using the dual mode configuration. The particle sensing unit was enclosed in order to protect the electronic circuitry, having overall dimensions of 49 mm × 44 mm.

B. Solidly Mounted Resonator Structure

Zinc oxide based solidly mounted resonators were used as the sensing element for developing the particle detector. A schematic of the employed SMRs is shown in Fig. 3. The SMRs were fabricated on a p-type Si (100) substrate and consisted of a 2.96 µm thin film of ZnO sandwiched between 200 nm thick Al electrodes and deposited on top of an acoustic mirror formed by three pairs of alternating layers of 1.82 µm Mo and 1.65 µm SiO_2. The c-axis oriented ZnO layer was reactively sputtered from a 4-inch Zinc target with an Ar/O_2 mixture using a high target utilisation sputtering (HiTUS) system [28], obtaining a deposition rate of ~20 nm/min.

The SMRs were fabricated using a 4 mask photolithography process for the patterning of the acoustic mirror, the bottom electrode, the top electrode and opening via through the piezoelectric. The sensing area of the device determined by the overlapping of the bottom and top electrode is 200 µm by 200 µm while the footprint of a single device is 1 mm square. The SMRs resonate at a frequency of ~ 970 MHz. Details on the design, modelling and fabrication of these devices are reported elsewhere [29].

An electrical signal applied between the electrodes generates a mechanical wave that propagates along the bulk of the piezoelectric material. The total mass of the particles deposited on the active area of the sensing SMR produces a shift on the resonant frequency, which needs to be measured.

These SMRs have been designed to operate in a longitudinal mode, which is characterised by particle displacements in the same direction of the wave propagation. The application of an alternating electric field between the two electrodes produces a longitudinal deformation through the thickness of the piezoelectric material in a thin film resonator. Upon deformation, the acoustic wave propagation through the bulk of the material across the crystal and the particle displacement are both in the same direction, normal to the sensor surface. Longitudinal mode SMRs have been used in this work for nanoparticle detection as they have been found to be providing promising results for sensing in air or gas [30, 31]. A constructive interference between incident and reflected bulk waves occur when the wavelength is an odd multiple of the double substrate thickness. Thus a standing wave is created inside the sensor boundaries as illustrated in the schematic drawing shown in Fig. 4.

C. Oscillator Circuitry and Interface Board

Acoustic wave perturbations induced by particle deposition present themselves as attenuation and velocity changes of the bulk wave; however, the real-time detection of these changes require complex and bulky circuitry unsuitable for integrated systems. It is possible to monitor the acoustic velocity changes indirectly with great measurement precision [32] by using the SMR sensor as a resonating element inside a simple oscillator circuit. The dual SMR resonators, both operating at a frequency of ~970 MHz utilized the Colpitts oscillator configuration with a grounded base configuration in order to obtain good frequency stability and sensitivity. Here the SMR input port is connected to the transistor's base and the output port is connected to the ground.

The Colpitts oscillator was chosen because it allows the SMR to operate in a 1-port configuration by grounding the output port as opposed to a Pierce oscillator in which it requires a 2-port configuration [33]. These oscillators offer good stability at frequencies above 500 MHz, lower harmonics, lower component count and hence lower cost than other types of oscillator circuits [34]. Fig. 4 shows a schematic of our oscillator circuit. The commercial NPN Silicon RF Transistor BFR92P used to provide gain to the active oscillator part, reduces the effect of parasitic capacitances considerably when compared to an op-amp, allowing the circuits to operate at higher frequencies. The resonator behaves like an inductor between the series and parallel resonance regions of the SMR device. The radio frequency (RF) transistor along with the feedback capacitor C4 provides the gain to compensate for the resistive losses in the resonator [34].

For the circuit to oscillate, the Barkhausen criterion needs to be satisfied when the SMR is connected between the RF transistor base and ground. The LC tank circuit and the grounding conditions of the transistor base provided through the SMR create the initial startup oscillator frequency, which is a few MHz above the steady state frequency. The LC oscillation noise and the wideband noise energy at the SMR´s resonance frequency will get stored in the device [35]. The following two conditions including unity total loop gain magnitude and 0^o phase shift for the entire loop [36] will be fulfilled when a standing electrical wave is created inside the resonator. As the energy builds up in the SMR, more current flows through the device, which results in shifting of the oscillation frequency to the resonance frequency of the SMR. The output spectrum from the Colpitts based SMR oscillator showing a resonant frequency of 933 MHz obtained by an RF oscilloscope (Tektronix MDO3012 Mixed Domain Oscilloscope) is shown in Fig. 5.

When the particles are deposited on to the sensor surface, the total phase of the feedback loop will get shifted by a certain amount due to attenuation and velocity changes of the bulk wave. As a result, the Barkhausen criterion will be satisfied at a lower frequency and thus the resonance of the SMR will change from the initial resonant frequency to a lower resonant frequency, resulting in a shift in frequency. In order to measure the frequency shift caused by the particles deposited onto the sensing SMR device, an interface board was designed that consists of a double balanced RF signal mixer (Hittite Microwave Corporation), an RF low pass filter (Mini-circuits®), a comparator (Analog Devices) and a dual linear voltage regulator (Micrel®). The oscillator boards were connected to the interface board and powered by the 2.5 V output of the low-dropout regulator. The reference and sensing oscillator frequencies were mixed using a heterodyne down-conversion technique and the low pass filter was used to output only the difference frequency. In this way, the mixing circuit helps in reducing both the effects of common mode variations and the output frequency signal range. The comparator converts this differential frequency output into a digital signal with voltage level compatible to the voltage tolerance of the microcontroller digital input pins (3.3 V), where the frequency counting takes place. The interface board is powered by the 5 V supply of the microcontroller, which in turn is powered via a USB connection to the PC.

A low-cost microcontroller Teensy 3.1 was used in order to accomplish the frequency counting of the square output signal and log the data to a computer through USB serial communication. These data were recorded with virtual instrument developed using LabVIEW software which also allows the real time visualization of the data.

III. EXPERIMENTAL SETUP

Characterization of the developed particle sensor based upon solidly mounted resonators took place inside a sealed environmental chamber at VITO, Belgium. A schematic of the experimental setup is shown in Fig. 6. The SMRs were placed inside the test chamber together with a range of reference commercial instruments for real-time monitoring of particle deposition within the test chamber. The commercial sensors include an acoustic based Quartz Crystal microbalance (Vitrocell® Systems), and two different optical particle counters (Dylos Corporation, Grimm Technologies Inc.), which were placed adjacent to the SMR based research sensors.

The test rig consisted of a dust generator (TOPAS®), a suction pump for controlling the dust flow into the chamber and a humidity control unit. Photographs of the experimental setup are shown in Fig. 7. Typical conditions inside the chamber with an internal volume of 0.72 m^3 were 24°C and 22% RH. The SMR sensors and the commercial instruments were placed at one of the corners inside the chamber as shown in Fig. 8. The PM concentration readings from all the instruments were continuously logged to the PCs.

The target particles during these measurements included Arizona test dust (Powder Technology Inc.) with nominal particle diameter of 0-3 µm. The particle size distribution of these test particles had a median, d_{50}, of 0.927 um. A 90% of the particles were found to be below a diameter of 1.526 um (d_{90}) whereas only 10% of the particles were below 0.712 um (d_{10}). The free-settling velocity [37] of Arizona dust particles with 1.5 um in size would be ~81 um/s whereas finer particles with a diameter of 0.5 um fall at ~9 um/s. Therefore, only the bigger particles will fall onto the resonator whereas the very small particles are unlikely to settle on the device. Hence, UPFs are not detected with this method.

Particles were injected into the chamber for a certain period of time ranging from a couple of seconds to up to 10 minutes obtaining dust concentrations as low as 20 $\mu g/m^3$ and as high as

30,000 µg/m^3 as measured by the optical particle counters (OPCs); namely the Grimm monitor. The longer the particle injection period, the greater the amount of particles inside the chamber. As the particles are injected, the commercial optical sensor draws a sample air and measures the particle concentration inside the chamber in its output units (µg/m3). On the other hand, as gravitational sedimentation is used to collect the particles onto the SMR-based sensor and the commercial QCM device, the sensors will show a frequency shift only after particles have settled on its surface. The greater the amount of particles that were injected, the greater amount of particles that will deposit onto the resonator.

IV. RESULTS AND DISCUSSION

After the injection of the test particles into the environmental chamber, a change in the oscillating frequency of the SMR based sensor was observed. Fig. 9 shows a typical frequency shift measurement of the SMR system when exposed to a predefined amount of dust concentration. There was a decrease in the resonant frequency of the SMR sensor as shown in the fig 9, due to the addition of nanoparticles on to the sensors. The environmental chamber was completely sealed to ensure ambient temperature stability, to avoid any external wind effects and the deposition of any foreign material onto the sensors within the laboratory setting. Additionally, the effects of any temperature or humidity changes are also eliminated by operating the sensor system in the differential mode.

As the deposition method is based on the sedimentation of the particles due to gravity, an average particle settling period of 2 minutes was observed for Arizona dust defined by the time in which the frequency of the SMR sensor started shifting due to the added mass of the settled particles. The real time measurements of the SMRs are comparable to the response of the commercial QCM device. The operating frequency of both acoustic wave based devices (SMR and QCM) decreased due to the added mass of particles falling onto their sensing surface at very similar times. However, the mass sensitivity demonstrated by the SMR device is orders of magnitude higher than the reference QCM sensor (thousands of kHz for the SMR compared to less than 1 Hz for the QCM) as can be noticed from Fig. 10.

The mass deposited at the surface of the SMR sensor follows the thickness extensional vibration of the piezoelectric material and hence the loaded SMR would simply behave as if it were thicker. The effective wavelength of the bulk wave is thus increased and consequently its resonant frequency decreases. This explains the frequency shift produced by the deposition of nanoparticles on the active area of the solidly mounted resonator, assuming that there is no energy dissipation and hence will only be valid for thin, rigid and uniform films [37] having similar acoustic behaviour as that of the bulk piezoelectric material.

However, when the acoustic properties of the deposited viscoelastic nanoparticle probe layer differ significantly from those of the SMR, it experiences viscous coupling primarily due to the lossy surrounding media causing the shear longitudinal waves to dissipate into the adjacent media, resulting in the degradation of the Q-factor. The longitudinal shear wave propagating into the adjacent lossy medium depends on the resonant frequency of the SMR and the signal amplitude is degraded exponentially with a characteristic decay length (δ) [38, 39] that is given by (1):

$$\delta = \sqrt{\frac{2\eta}{\omega\rho}} \qquad (1)$$

where $\omega = 2\pi f$ is the angular frequency, η is the viscosity and ρ is the density of the lossy adjacent medium.

The frequency response of the SMR particle sensor was measured to various levels of dust concentrations to establish the sensitivity of the particle sensor. Based on the data collected from the experimental measurements, the relationship between the SMR sensitivity and particle concentrations has been found to be a linear response as shown in Fig. 11, with a sensitivity of 4.6 Hz per µg/m^3 confirming to have the SMR sensor to be operating in the non-saturated regime. In addition, the minimum detectable particle concentration of the SMR based system was found to be about 20 µg/m^3.

High frequency solidly mounted resonators will only respond to the surface interactions that occur within the close proximity of the sensor surface. An 870 MHz SMR based sensor will have a decay length that is up to the range of 1 µm, depending on the type of the adjacent lossy medium probe. As a result, those particles that are having diameters smaller than the decay length, will only get probed by the SMR device by allowing acoustic coupling of the entire particle volume. Otherwise, the sensor only probes the particle partially near its surface, resulting in the non-response of the sensor due to larger sized particles. Hence there is a need for tailoring of the decay length to suit the size of the particles to be detected.

The decay length of an acoustic wave resonator device always depends on the sensor resonance frequency. For an SMR device, the amplitude and decay length of the longitudinal acoustic wave transmitted to the adjacent lossy medium decrease with increasing sensor resonance frequency. This enables the particle sensor described here to be capable of detecting submicron-sized particles with picogram mass range, using sensitivity-tailored frequency-dependent designs of acoustoelectric sensors. During the performed experiments, we haven't observed saturation of the sensors which suggest that the particles may have not form multilayers on the resonator surface.

V. CONCLUSIONS AND FURTHER WORK

A particle sensor has been developed based upon two solidly mounted resonators interfaced to Colpitts type oscillators. The sensor works in a differential mode compensating for common temperature and humidity effects. The SMR based system was characterized inside an environmental chamber were particles of 0-3 µm in size were injected. The added mass of the particles deposited on the resonator caused a frequency shift on the SMR device. Real-time measurements of the frequency shift demonstrated the capability of these acoustic wave based devices to detect fine particles with a sensitivity of 4.6 Hz per μg/m^3. Particles were collected onto the resonator surface by gravitational sedimentation. However, by using this method ultrafine particles are unlikely to settle on the device. Therefore, for the development of the full sensor system other particle deposition methods will have to be implemented. Further work is currently being carried out towards the development of a low-cost particle sensor system in a package (SiP) based on SMR devices by interfacing the resonators to standard CMOS circuitry. Ultimately full integration of the SMRs with CMOS technology will be realized for the development of a low-power, low-cost smart miniature sensor for the real-time monitoring of particulate matter.

ACKNOWLEDGMENT

We acknowledge Prof. Andrew Flewitt and Mr. Girish Rughoobur at Cambridge University for fabrication of the SMR devices. Miss F.H. Villa-López thanks the financial support from the National Mexican Council of Science and Technology (CONACYT).

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Biographies

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This article has been accepted for publication in a future issue of this journal, but has not been fully edited. Content may change prior to final publication. Citation information: DOI 10.1109/JSEN.2015.2512303, IEEE Sensors Journal

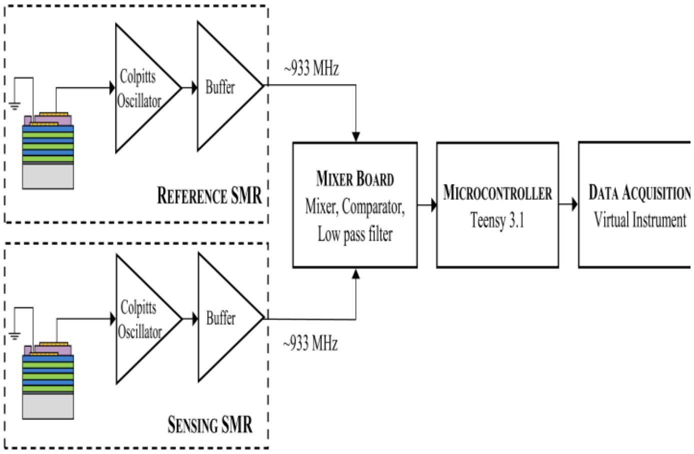

Fig. 1. Overall structure of the developed differential mode particle sensing system based on Solidly Mounted Resonators.

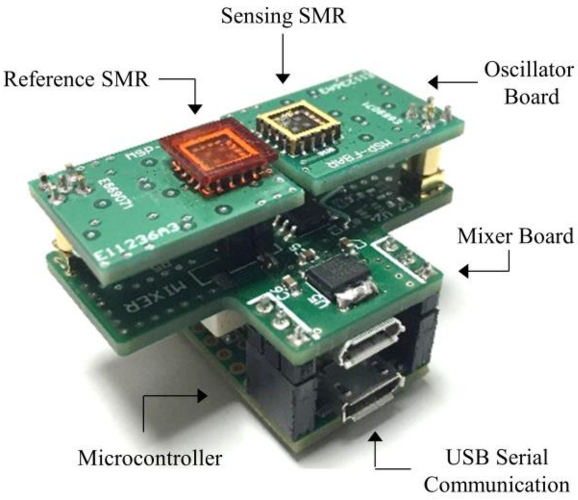

Fig. 2. Photograph of the developed particle sensing unit using SMRs and working in a dual configuration. The overall dimensions are 49 mm × 44mm.

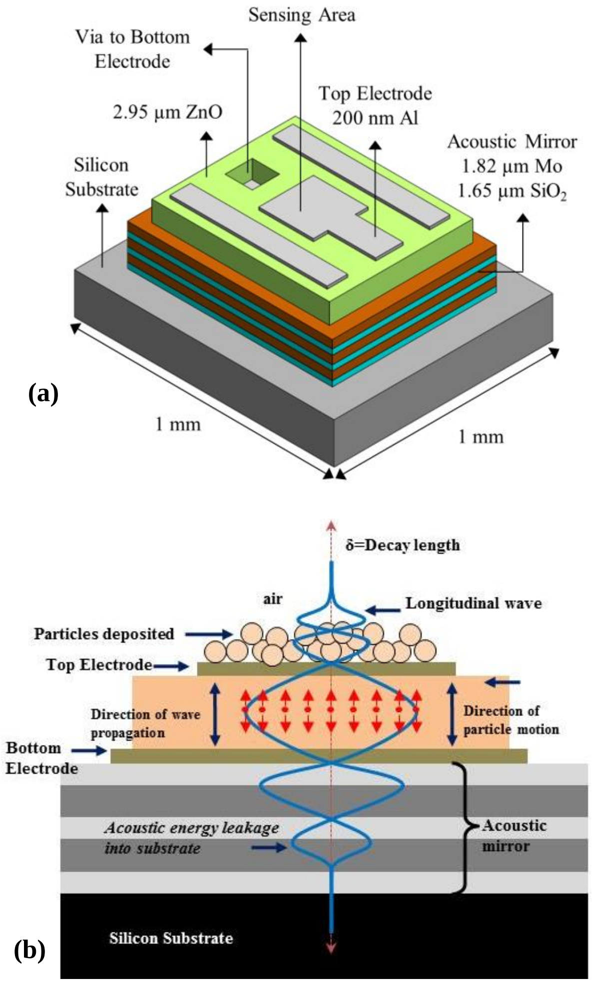

Figure 3 *(a): Three-dimensional* representation of the ZnO based Solidly Mounted Resonator with Al electrodes operating at ~970 MHz*; (b):* Schematic diagram showing the longitudinal bulk wave propagation inside an SMR sensor illustrating the standing wave pattern created inside the resonator thickness and the decay length into the adjacent medium associated with the bulk wave.

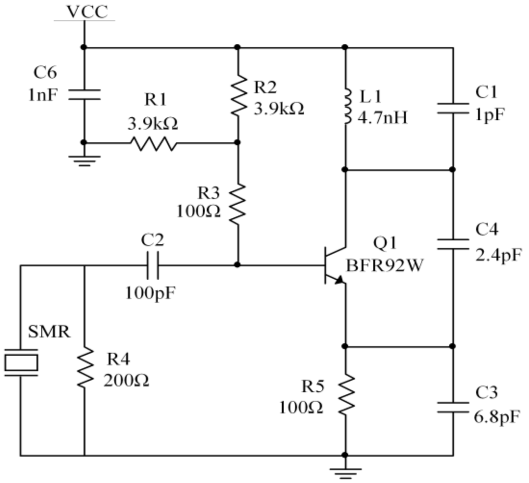

Figure 4. Schematic of the Colpitts type oscillator circuitry designed for driving the SMR devices.

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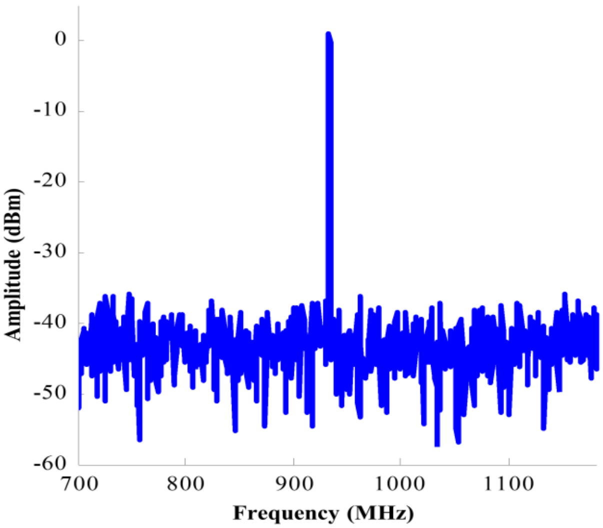
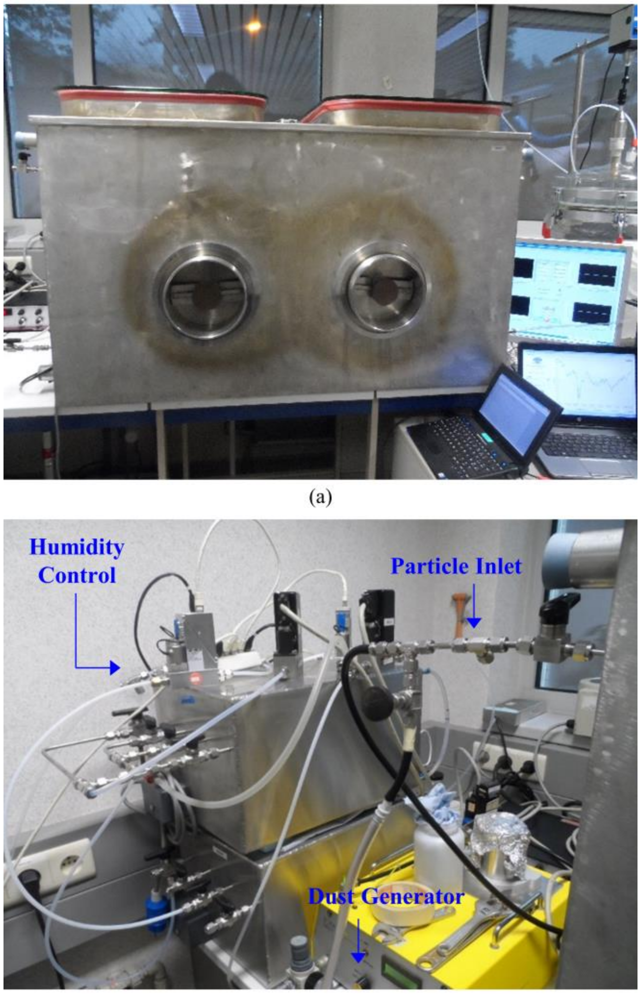


Fig. 5. Spectrum of the SMR-Colpitts oscillator showing a resonant frequency of 933 MHz.



(a)

(b)

Fig. 7. Experimental setup: (a) Environmental chamber with data acquisition hardware and (b) humidity control and dust generator setup.

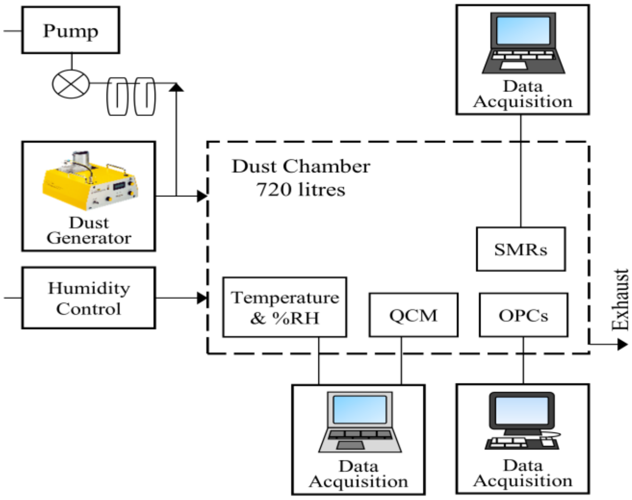


Fig. 6. Block diagram of the setup used to perform particle testing with the developed SMR-based unit.

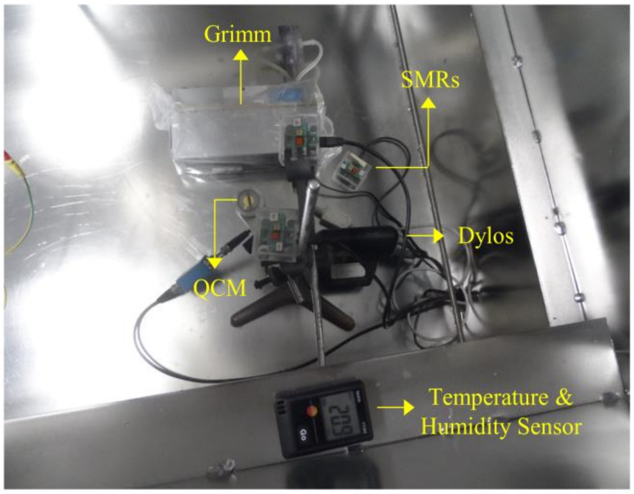


Fig. 8. Experimental setup: SMR based particle detector and reference instruments inside the test chamber.

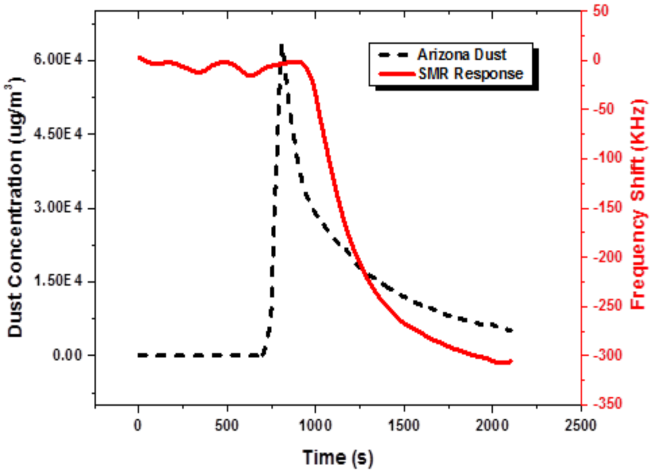


Fig. 9. Frequency shift of the SMR particle sensor due to the injection of a known concentration of Arizona dust

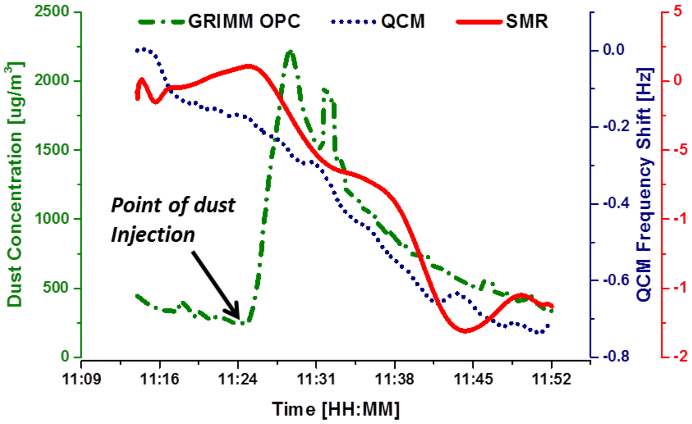


Fig. 10. Real time frequency shift measurement of the SMR sensor response to the deposition of Arizona dust compared to the commercial monitors including QCM device and Grimm OPC.

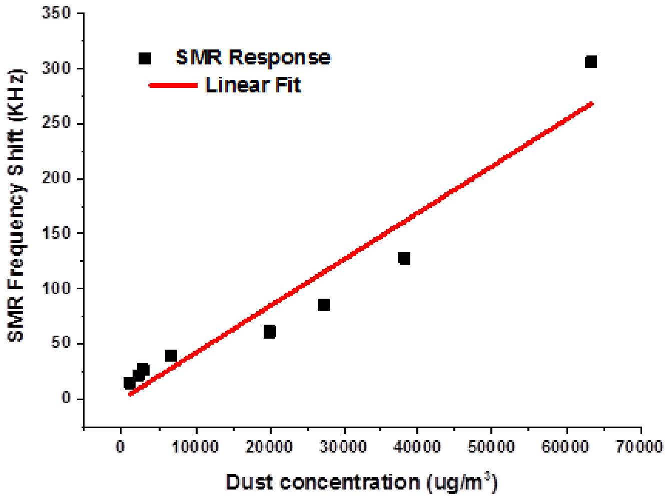


Fig. 11. Measured frequency shifts of the SMR sensor due to the different concentrations of Arizona dust.