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UNIVERSITY OF CALIFORNIA
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Experimental Investigation of
Mass Sensing With
Surface Acoustic Wave Devices

A Dissertation submitted in partial satisfaction
of the requirements for the degree of

Doctor of Philosophy

in

Mechanical Engineering

by

Frank Dickinson MacDonald

December 2010

Dissertation Committee:

Dr. Guanshui Xu, Co- Chairperson

Dr. Junlan Wang, Co-Chairperson

Dr. Javier Garay

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2010

The Dissertation of Frank MacDonald is approved:

Committee Co-Chairperson

Committee Co-Chairperson

University of California, Riverside

ABSTRACT OF THE DISSERTATION

Experimental Investigation of
Mass Sensing With
Surface Acoustic Wave Devices

by

Frank Dickinson MacDonald

Doctor of Philosophy, Graduate Program in Mechanical Engineering
University of California, Riverside, December 2010

Dr. Guanshui Xu, Chairperson
Dr. Junlan Wang, Cochairperson

We present an experimental study of mass sensitivity for a Surface Acoustic Wave (SAW) sensor. Chemical SAW sensors have been developed to detect the mass variation of the mass adsorbed into sensing film by showing resonant frequency shifts. Previous experimental results have focused on the static sensitivity of theoretical SAW sensors assuming that the film thickness is negligible compared to the central wavelength of surface waves. Most current research is focused on dynamic experimental measurement and analytical techniques that are application specific. We have studied a SAW sensor with relatively thick isotropic film layers sputtered on as a static measurement comparison. The results can be used in

a detection algorithm to quantify mass on the SAW sensor. Our results show a set of nodal response wave form changes and frequency shifts due to a continuous mass distribution. The development of the static experimental technique was completed with duplicate SAW tests. The duplicate SAWs had 12 measurements with 10 independently sputtered layers of 100 nm SiO₂, hermitically sealed, and hermitical seal removed using a HP8510A network analyzer. In addition, our results show worst case total error of 0.1% between replicates. Further testing and modeling is required to correlate macro to micro scales for quantifying mass detection with SAW sensors. This research discusses the issues of utilizing a SAW sensor that could be readily integrated into cell phones for distributed sensing as being requested by Homeland Security.

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1. INTRODUCTION

1.1 Abstract

Surface Acoustic Wave (SAW) sensors support the need for portable, robust and wireless detection of toxic industrial chemicals, chemical and possibly biological agents. Chemical detection with SAW sensors is extremely varied in technique and approach. Notch filters are the most robust SAW sensor designs currently being utilized. Acoustic wave chemical detection shows promise that is just beginning to be realized in commercial products. Competition from alternative technologies is also advancing rapidly towards commercial reality. Currently, the research performed on all detection technologies has been focused on developing reliable low cost concepts. Effort to address sensitivity, accuracy, repeatability and life time performance of acoustic based sensors has not been performed in lieu of rushing products to market.

This dissertation will briefly review what a SAW sensor for chemical detection is and sources of variability impacting accuracy and repeatability. This dissertation will discuss current research results related to minimum detection limit relative to an HP 8510A network analyzer. As well, this dissertation will explore the possible use of SAW sensors with more complicated response in chemical detection and identification process.

1.2 Overview & Focus

Micro sensors are an area of research interest focused on improving sensitivity and reliability. The chemical sensor is required to quickly and accurately detect and identify in real time, a wide range of toxic chemicals at very low levels in gases. There are numerous applications for chemical sensors, including environmental monitoring, rescuer safety, production process engineering, transportation engineering, and Homeland security.

Consequently, researchers are becoming more interested in real-time detections of hazardous pollutants and chemical agents in the air by remotely operating sensors rather than using time-consuming conventional analytical laboratory methods. Sensors based on surface acoustic wave (SAW) devices have shown considerable potential for these applications because their direct response to physical and chemical parameters, including surface mass, stress, strain, liquid density, viscosity, permittivity and conductivity. In addition, the miniaturized size, low production cost and wireless accessibility also contribute to its advantages.

Prior analytical efforts by Xu and Wang [44] in 2006 produced a numerical model for the analysis of sensitivity of SAW sensors. Due to near surface computational limitations, a finite element model was employed to understand the effects of film modulus and thickness. Second order mass loading effects resulted in significant time delays for SAW sensors coated with relatively soft films. An increase in film density was

predicted to prolong the time delay and positive frequency shifts were predicted for non-conductive thick films. Opposite directions of frequency shifts for conductive films were obtained. The numerical model predicted that thicker and softer films lead to SAW sensors with better mass sensitivity. As well, several electrical coupling models exist for SAW transducer design. The applicability of the model application to improve SAW sensor design understanding needs to be experimentally determined.

Establishing this experimental capability resulted in the need to develop a test method and comparing to other research on film properties that include insulating, conducting and adsorption. Literature papers lead to concepts of signature analysis to improve sensitivity & identification, and reversible adsorption thin film with Zeolite. As well, acquiring for research purposes a commercially produced SAW sensor identified the limited production costs as an issue. Further research identified other researchers at University of Florida as utilizing alternative central frequencies and mass manufactured SAWs for other markets which reduced costs by two orders of magnitude. The mass manufactured SAW market evaluation exposed the central frequency and cost as major parameters that needed evaluation for SAW chemical detection sensors.

1.3 Background

The primary mechanism of SAW chemical sensors is to detect the frequency shift and attenuation loss caused by the absorption of chemical agents on a thin polymer film coated on the surface of the SAW device. These devices have been successfully fabricated and tested. However, hermetic sealing is used to eliminate environmental and contamination variability in everyday electronic circuitry appliances

such as cell and mobile phones, and televisions. Since hermetic sealing is removed in SAW chemical detection, radio frequency identification markers are utilized for detection with a hermetically sealed SAW as reference to adjust for environmental variables. However, a hermetically sealed reference SAW is unable to correct for component short term and long term drift mechanisms, such as moisture, pressure, dust or other interferants, affecting sensitivity and selectivity of this detection process. Combined orthogonal detection techniques are being pursued as a means of addressing system short term drift, but are unable to detect long term drift without a standard and calibration process that provide zero referencing of the sensors.

The use of a Network Analyzer (NA) to study frequency shift of SAW sensor vs. impedance change for a piezoelectric resonator near resonance is an established technique. As well, NA systems are utilized to fully characterize electrical circuits in the radio frequency domain above 10 MHz. A NA device is a discreet measurement device meant to be a quantitative device traceable to original equipment or national standards.

Recent work has focused on commercialization of SAW detection devices based on the circuit for a SAW oscillator using a resonator or delay line application. Inherently, these measurements are different by being continuous qualitative measurements as the adsorption into the film changes the applied mass which results in a frequency shift. The films are applied to the bare SAW with a spin coating technique where a drop of the material is spun to uniformly spin to the edges of the device. There are numerous SAW designs which will be overly simplified here and referred to as resonators or delay lines.

Some SAW analytical research has been focused on delay line applications with the thin film placed between the interdigital transducers. Due to the surface acoustic wave existing within one wave length of the surface, numerical techniques are excessively computationally intensive which leads to simplifying assumptions being utilized to solve these types of problems. Since the models are being developed to predict sensor design optimization approaches, experimental understanding is required to validate assumptions. Linear perturbation is a common assumption utilized to simplify the wave problem without translation into the application of damping vs. signal amplification that limits the accuracy of the devices being developed for commercialization.

Adsorption onto different films with selectivity differences is used as a way of identifying the chemical in a multiple SAW sensor application. The multiple sensor SAW sensor utilizes a detection algorithm to identify the chemical. Further, simplifying assumptions that all adsorption is reversible, evenly distributed, and follows a constant analyte concentration are commonly made for a uniformly adhered film. The adsorption assumptions are not studied often due to the sensors making dynamic measurements. Martin et. al showed cumulative frequency response to vapor diffusion as function of time in 1987 which is widely utilized in the sensor systems. The complexity of a static measurement (RF shielding, phase & attenuation changes, lack of relevant physical properties for film, and environmental variations) and comparison to bulk chemical samples explains why quantitative measurements have not been made to check the assumptions.

2. HISTORICAL VIEWPOINT

2.1 Review

The piezoelectric effect was discovered by Pierre Curie in 1883. It took another 80 years for detailed scientific understanding to start to occur after 1950 even though Mueller first studied Rochelle salt in 1935. The development of radar and the need for a faster chirp was the motivation for this development during WWII. However, the telecommunication industry has been the largest consumer (~3 billion/year) over the last few decades of surface acoustic wave (SAW) devices in cell phones and base stations. The communication SAWs act as bandpass filters in both the radio frequency and intermediate frequency sections of the transceiver electronics. Sensor applications are the emerging market for acoustic wave technology in medical, automotive, integrated chip manufacturing, gas or chemical handling & safety, environmental monitoring, rescuer safety, production process engineering, transportation engineering, and Homeland security.

The current motivation for acoustic sensors is based on the possible use of chemical or biological weapons against military or civilian targets. The sensor is needed to rapidly detect harmful agents, correctly identify the agent and define the area of exposure. Rapid detection is required so that a threat can be recognized and protective gear donned (ideally in seconds based on Mission Oriented Protective Posture (MOPP) level). It is also important to know the extent of contamination. During the Tokyo

subway sarin attack in 1995, 9% of emergency medical services (EMS) providers suffered the affects of acute exposure.

A large variety of sensor types are available to identify liquid droplets of chemical agents on surfaces and in vapors. However, sensors are currently challenged with obtaining an appropriate sample for analysis and filtering out nonhazardous environmental chemicals that may be present. The laboratory-based equipment can also detect agents in water; however, laboratory-based equipment is cumbersome or incapable to perform tests in the field. This dissertation is focused on SAW chemical sensor, which has been extensively studied as shown in Reference [1] to [17]. Figure 1 is an example of the many commercial attempts to develop SAW chemical sensors over the last few years.

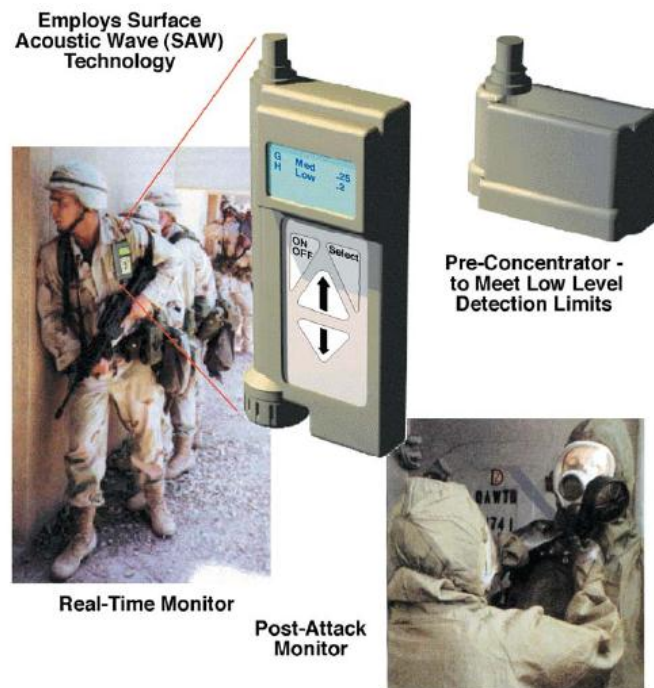


Figure 1. Military SAW Chemical Detector.

2.2 Overview

Acoustic wave technology generates waves utilizing the piezoelectric effect originally found in materials such as Quartz, Rochelle salts, or ceramics. The piezoelectric effect is a deflection induced by a voltage across the material or a voltage created by a deflection across the material [1-6, 20, & 33] & [w1-w20]. Essential to understanding dielectrics is through capacitance. Capacitance is based on the ability of two conductors to store a charge. The capacitance of two conductors separated by a vacuum is described in Equation 1 and shown in Figure 2 [w1 & w5]. The way to increase energy storage other than increasing the plate size is by inserting a dielectric material between the plates. The dielectric material has randomly oriented atoms that when a charge is present reorient/stretch as shown in Figure 3 [w5]. It is this reorientation that allows the material to increase the capacitance (Equation 2) and induce deflections. The dielectric constant is a property that determines its ability to become electrically polarized. The higher the dielectric constant, the more charge that can be stored which allows the creation of smaller electronic circuits. This voltage and deflection coupling is the response that drives a lot of technology. The SAW circuit modeled in a capacitance sense provides the macro scale viewpoint satisfying Ohms law.

$$C_0 = Q/V = \epsilon_0 A/d \quad (1)$$

$$C = \frac{\epsilon}{\epsilon_0} \frac{A}{d} \quad (2)$$

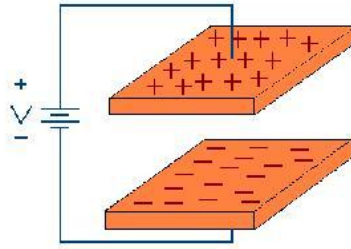


Figure 2. Diagram of the basic capacitor structure.

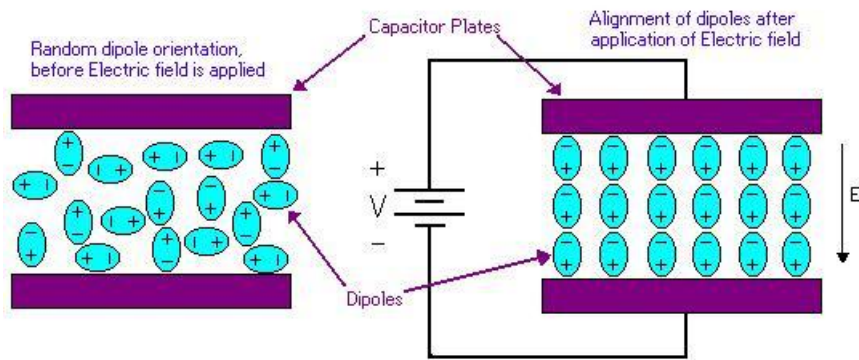


Figure 3. Orientation of dipole moments.

An acoustic sensor works on an oscillating electric frequency transmitted between two interdigital transducers (IDT) separated by a distance via a piezoelectric material. The first electrode (IDT1) is working as a transmitting transducer, which converts an electrical signal into a surface acoustic wave. The other electrode (IDT2) receives the acoustic wave and transduces it back into an electrical signal. If a SAW device is coated with a thin chemical selective polymer film between the IDTs, it will cause an insertion loss and a frequency shift to the output signal. If the polymer coating is exposed to chemical vapors that can be adsorbed into the polymer materials, it will change the mass loading and elastic properties of the film, thus cause a further insertion loss and

frequency shift. The micro scale viewpoint is focused on the IDTs coupling with the piezoelectric material to minimize loss.

Optimization of SAW sensor performance is performed by adjusting the physical dimensions of the IDT and spacing between the electrodes while considering what wave speed the piezoelectric material can sustain. Because SAW sensors have most of the acoustic energy contained in 1 wavelength of the surface, they have the highest sensitivity of the various acoustic sensor types. Normally, SAW devices are packaged in hermetically sealed cavity style packages to insure that their performance will not change due to a substance (moisture, dust, etc.) contacting the surface. The SAW sensors use this sensitivity to detect chemical or biological agents with a thin absorbent material attached to the surface between the IDTs as shown in Figure 4 and discussed in [14-20]. By comparing the response of a hermetically sealed SAW to a detecting SAW, a relative comparison of the mechanical strain affecting propagation path length and surface wave velocity can be made to perform pattern recognition to identify the absorbed substance. Use of prior test data to justify the pattern recognition process is time consuming and prone to shifting from environmental variables in nonlinear coupled ways. Hence, the general approach for agent identification is to tailor the film for selective absorption while putting multiple SAWS with different coatings in parallel and use of a relative slope change in frequency to identify the presence of agent. The identification of particular agent present is still difficult because of relative change and no absolute measurement. The changes in frequency and/or phase correlate with surface strain via

Equation 3 shown below which shows promise for the possibility of absolute measurement. The above discussion is discussed in great detail in References [7-14].

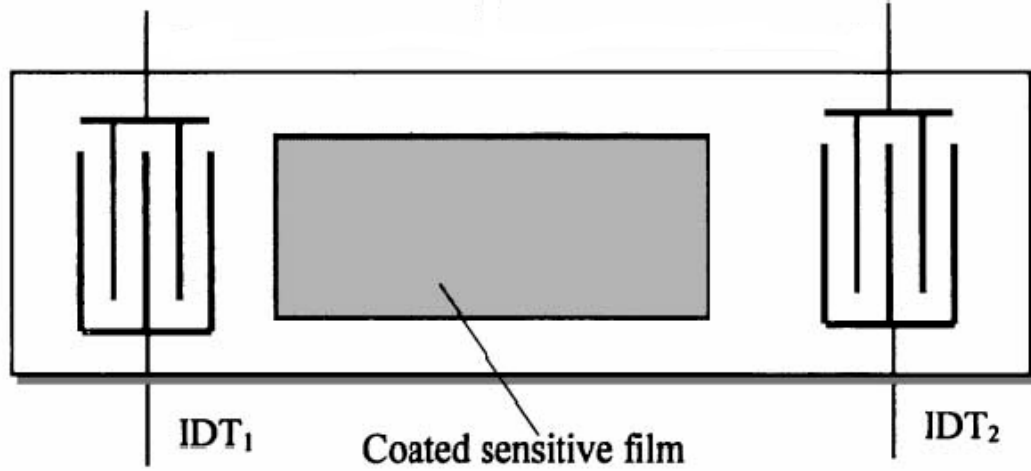


Figure 4. A typical layout for a SAW chemical sensor.

$$\sigma_1 = \frac{1}{S_{11}^E} * S_1 - \frac{d_{31}}{S_{11}^E} * E \quad (3)$$

where:

- σ_1 = stress in the beam length direction
- E = electric field
- S_1 = elastic strain
- S_{11}^E = elastic compliance & fixed electric field
- d_{31} = piezoelectric coefficient

The most common acoustic wave sensor material is single crystal quartz (SiO_2). Temperature effects can be significant based on crystal selection, cut angle and propagation direction. Other common acoustic wave sensor materials include lithium tantalite (LiTaO_3), and Lithium Niobate (LiNbO_3). Generally, single crystal quartz is selected to minimize temperature effects and cost. Other piezoelectric ceramics

commercially available are Alumina (Al_2O_3), toughened Alumina Titania (TiO_2), Gallium Arsenide (GaAs), langasite (LGS), silicon carbide (SiC), Zirconia (ZrO), and Zinc Oxide (ZnO). Microelectronic mechanism system (MEMS) applications motivate the use of these materials and/or increasing operating frequency above 1 GHz for telecommunication, radar, medical scanning purposes. Other specialty dielectric materials commercially available include Lead Zirconate Titante (PZT), polyvinylidene fluoride (PVdF), polymer sheet, and aerogel. PZT is a material created by compressing a polycrystalline mix under high pressure and magnetic field to create a polarized material. PZT will oscillate at more than one frequency based on voltage and current levels, which shows promise for a tailorable SAW at the cost of operating life and expense. The above materials discussion comes from References [8-9, 13-14, 20 & 33]

Wave guide materials include PZT, PVdF, polymer sheet and aerogel that are being researched for thin film dielectric applications for advanced integrated circuits. The motivation is driven by organic, inorganic and hybrid materials that allow the IC device manufacturer to meet desired device speed increase and reduced power consumption while migrating to the larger 300mm wafer size. Further, the use of spin-on dielectrics to lower k values allows low shrinkage characteristics that make them very stable when coated sequentially, with no need for intermediate furnace cures. These features allow cost effective, high throughput, Low-k stack deposition processes. These same features may allow design flexibility in SAW chemical sensor design [w21].

Acoustic-electronic and acousto-optic components for signal processing motivate the need to understand theoretical topics impacting wave generation and propagation.

Understanding the issues effecting SAW chemical sensor detection starts with detection level. Sensitivity (threshold) quantifies agent detection level. Hazardous chemical levels are based on concentration levels where as biological hazardous levels can be at the particle level. The quantity detection level defines the motivation for sensor developments in micro cantilevers, and enhanced raman spectroscopy that are focused on live biological specimens. An example is Aluminum Nitride nanomechanical flexural resonators being pursued as discussed in Reference [42], which reduce detection quantity for biological detection. While microelectromechanical system (MEMS) size is more appropriate for SAW chemical sensors, chemical detection is achieved by blowing filtered air over an absorbing polymer layer. SAW biological organism detection can be achieved at the particle level by suspending in a fluid and reacting with a tailored analyte. Besides sensor size being focused on critical quantity of agent, the sensor is designed to filter out nonhazardous environmental interferants that may be present. However, detection sensitivity is impacted by dynamic, static, or steady state agent concentration variation. A further complication from sensitivity variation is compounded by mechanical and electrical errors that will be discussed for SAW sensors.

Agent detection and identification result from repeatability in sensor sensitivity and accuracy. Repeatability in a SAW chemical sensor is limited by drift or stability in frequency shift from small amounts of agents. SAW life time performance and specific design susceptibility to operating environments (pressure, temperature) and interferants (dust, moisture, etc.) further exaserbate the quantity of interrelated issues that need to be addressed when designing a sensor. SAW chemical sensors have the added advantage

that they are not sensitive to handling loads (vibrations from bumps or blowing air). However, the polymer placed between IDTs is sensitive to thermal loading, which is used to reverse the diffusion process. In concise review, the list of issues that need to be addressed for sensor development are shown below.

Sensor Issues:

- Sensitivity (Threshold) effected by
 - Dynamic, Static, or Steady State
- Accuracy impacted by errors
 - Mechanical, Electrical
- Repeatability effected by
 - Drift or stability (shift)
- Life time performance
 - Cycling Life, Operating Life,...
- Performance in severe environmental conditions
 - Elevation, temperature, moisture, interferants

SAW chemical detection typically operates between 25 and 500 MHz. Newer applications are focused on increasing the frequency to increase sensitivity as can be understood from Equation (3) utilizing wave equation solutions as shown by Dr. Xin Wang [44]. Rayleigh waves are typically 5 orders of magnitude slower than other waves propagating in solids. The Rayleigh surface wave is typically designed to be 1 nm height with wavelengths 1–100 μm . It needs to be pointed out that the SAW sensor has the highest sensitivity of various acoustic wave sensor types, which will be shown later. SAW sensor manufacturing is very direct using a four step lift-off photo-lithographic process to generate aluminum IDTs on the surface of a quartz wafer as shown in Figure 5 [9]. The absorbing film can be put on using a spin coat or deposition technique to generate a thin coating between the IDTs. Hence, A SAW sensor has four major design selections that include choice of piezoelectric material, shape and size of device , shape

and size of IDT, absorbing layer material and size & shape. The choices for piezoelectric sensor configurations are shown in Figure 6 [w1, w16].

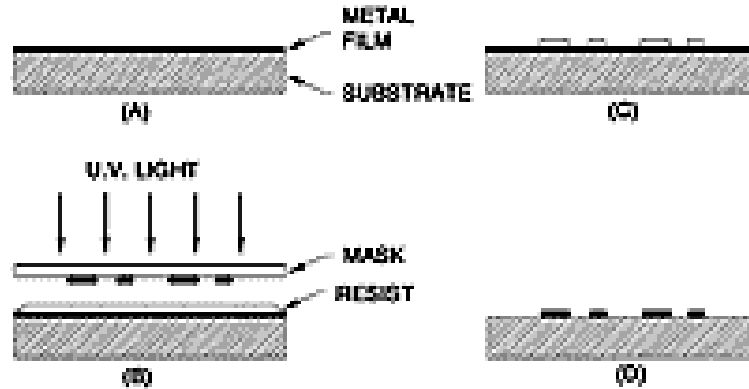


Figure 5. Photolithographic Process.

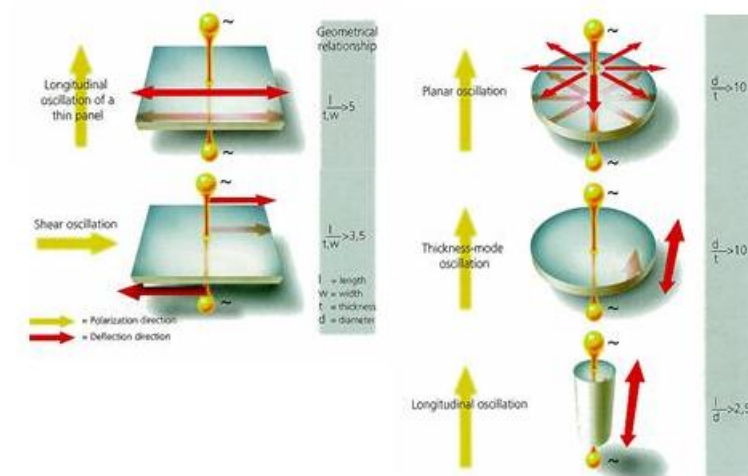
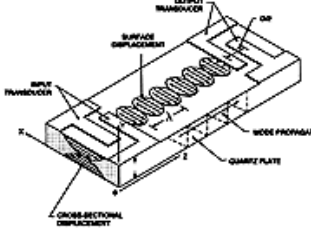
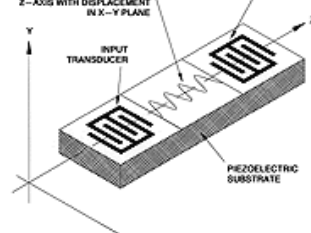
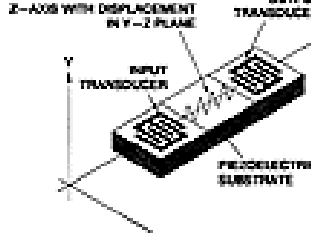
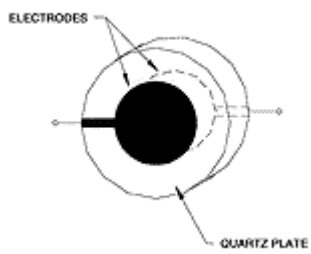
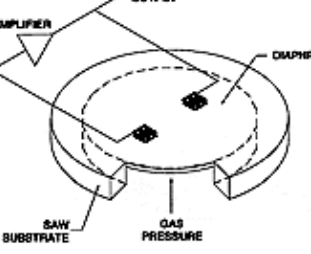
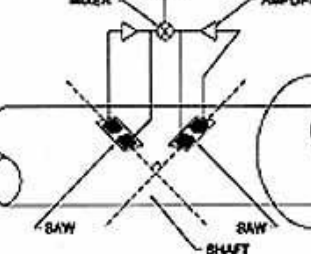


Figure 6. Fundamental dielectric response.

2.3 SAW Sensor Types

Many different acoustic sensor types are the product of dielectric response and design considerations that are beyond the purpose of this dissertation. However, SAW is the most surface sensitive while the most insensitive to flow and acceleration loads. The

liquid detection technique is primarily limited by reflection of acoustic waves. Types of sensor wave forms are shown in Figure 7 to 9. Applications of different sensors are shown in Figure 10 to 12, which can be better understood from References [9-26].

		
Figure 7. In the shear-horizontal acoustic plate mode (SH-APM) sensor, the waves travel between the top and bottom surfaces of the plate, allowing sensing on either side.	Figure 8. Shear-horizontal surface acoustic waves (SH-SAW) have a displacement that is parallel to the device's surface.	Figure 9. Rayleigh waves move vertically in a direction normal to the surface plane of a surface acoustic wave (SAW) sensor.
		
Figure 10. Although it is the oldest acoustic wave device, the thickness shear mode resonator is still used for measuring metal deposition rates.	Figure 11. The diaphragm flexes due to pressure, the SAW sensor changes its output.	Figure 12. The stress in the shaft is transferred to the SAW sensor, which changes its output frequency with stress and, therefore, torque.

2.4 Factors Affecting the Sensitivity

There are many factors that can affect the sensitivity of the SAW sensor. As mentioned earlier, a way of increasing sensitivity for detection is by increasing the operating frequency and/or sampling rate. The impact of increasing the operating frequency is best understood as discussed in [13-14]. Other researchers [7-8] are pursuing the sampling rates as well as flow control techniques to improve sensitivity of SAW chemical detection. Decreasing the film thickness and increasing selectivity can obtain further sensitivity improvements.

The process of vapor sorption into the polymeric films generally includes 4 stages [14] as shown in Figure 13: (1) Initial rapid adsorption; (2) Slower diffusion (absorption) into film with continual adsorption to resulting vacant sites; (3) saturation of the polymeric film with vapor molecules; and (4) multilayer buildup on the polymer surface. The frequency shift and insertion loss caused by the attachment of chemical agents on a thin polymer film coated on the surface of the SAW device is a mass driven response.

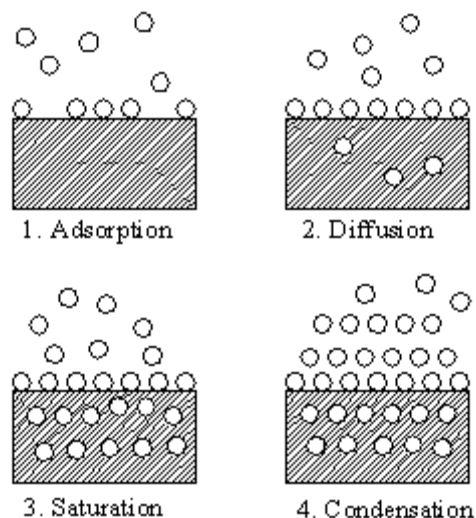


Figure 13. Stages in vapor sorption to a polymer film.

The majority of published descriptions of SAW chemical sensors for organic vapors exploit polymeric films of varying thicknesses and selectivity to provide the required degree of sensitivity. Siloxane and fluropolymer are polymers being used for thin film in SAW sensors that can be tailored with the addition of a chemically selective monomer [7-8]. The distribution of the monomer is expected to be distributed throughout the thin film at the cross link points for the polymer, which can be verified with differential scanning calorimetry (DSC). Dynamic mechanical analysis (DMA) is used to characterize the viscoelastic response of a polymer material, which may be used to verify hypothesis that will be proposed later.

Once the chemical vapor is transported to the surface of the coating on the SAW, the chemical adds to the mass of the coating via a transient non-uniform processes. Due to the abrupt change in the frequency response and insertion loss from the nonselective addition of mass, the resulting frequency change of several SAW's coated with different

thin films are compared to known patterns. It is this pattern recognition along with selectivity tailoring of the thin film that has been the focus of researchers over the last 5 years [7-19].

To reverse the process, clean air is blown over the SAW, which results in another abrupt frequency shift. Since most SAW chemical identification uses pattern recognition based on a relative change in frequency, it is generally assumed that all of the chemicals have been removed from the coating when the frequency shifts back close to reference SAW level. Logically, it can be realized that as the chemical diffuses out of the coating, the diffusion process slows down as discussed by Thompson and Stone in Reference [14] via $u_t = \alpha^2 u_{zz}$. This can be explained as the chemical has as much affinity to diffuse further into the coating as into the clean air once the process is reversed. Hence, it should be realized that once the coating has been exposed to a chemical, whether it is agent or interferant, the coating will not be clean without satisfying the diffusion equation based on time. As well, the repeated cycling of a chemical into and out of the thin film coating is physically aged which results in degradation versus time of the SAW detector. Physical aging effects can be reversed by going above the polymer Tg for a thermal plastic specific time (less so for a thermal set).

The structure of SAW device cannot eliminate the generation of bulk and shear waves besides the desired surface wave. Typically, a SAW is designed with damping materials to minimize reflections from these other wave forms in affecting the devices sensitivity. In combination with a wave guide and/or dynamic piezoelectric may be

utilized to minimize or take advantage of these waves, which are determined by the governing Hooke's law and the boundary conditions [3-6].

The operation environment will also affect the response of SAW chemical sensing device. It is well known that temperature can have a considerable effect on propagation velocity for acoustic wave devices, since many of the material constants involved are themselves temperature dependent. The temperature dependence of surface wave attenuation for quartz, in particular, has been studied over various temperature and frequency ranges [15-17]. Temperature can also have a considerable influence on film viscosity, stiffness and volume as has been studied in [18]. Pressure also contributes to acoustic wave attenuation and velocity change. In this case the attenuation is due to the generation of compressional waves in the gas in contact with the SAW device. This has been studied extensively by Slobodnik[40]. The attenuation was found to vary linearly with pressure. The significance of pressure effects is expected to be small, although original SAW pressure sensors were based on membranes.

2.5 Analytical Approaches

Due to the inaccuracies from relative frequency shift for agent detection, an analytical mathematical model is being developed and correlated with test data to address sensitivity issues in SAW chemical sensor design. Many analytic techniques have been developed in the past as best described by Reference [4] is expressed below. The approximate delta-function model (Tancell and Holland, 1971), the equivalent network model (Smith *et al.*, 1969), and effective permittivity model (Ingebrigtsen, 1969, Milsom *et al.*, 1977, Morgan, 1980) are particularly notable for their ability to capture the

principle effects involved in IDTs. Some more recent developments are the 2-D green's function (Huang and Paige, 1988), the coupling of mode method (Koyamada and Yoshikawa, 1979, Akcakaya 1987), the finite element method (Hasegawa and Koshiba, 1990, Yook-Kong Yong, 1997), and the boundary element method (Hashimoto and Yamaguchi, 1991). In spite of the certain strength of these analytical and numerical methods in addressing specific design requirement, they are generally unable to analyze the behavior of SAW devices on the full scale. None of them can predict accurate behavior of SAW devices for high frequency applications when several so-called secondary effects become significant (Jones *et al.*, 1972; Campbell, 1998). These effects include internal reflections in transducers, mechanical loading, bulk wave generation and reflection, and perturbation of surface imperfections.

The finite element method is presently the method of choice for the analysis of deformation and failure processes in solids. This method, in its present state of development, can be used to model an extremely broad range of physical phenomena, including electromechanical surface and bulk wave propagation in SAW devices. Modeling and simulation of micro devices have received ever-increasing attention from both mechanical and electrical engineers because they can be used as a design tool to enhance device performance and reduce the design cost. A model focused at the piezoelectric material coupling to the IDT was idealized by Xin Wang [44]. The solution shows the surface wave creation and propagation at a micro scale relative to the SAW device. However, modeling and simulation of SAW devices remain one of the most challenging problems. According to the authors knowledge, there has been no published

work regarding the direct transient finite element modeling and analysis of the SAW devices on the full scale as is proposed here. Effective small scale transient finite element models [3-6] tied with approximate full scale simulations utilizing capacitance or admittance models may provide the path towards full scale modeling that is required.

3. RESEARCH DESCRIPTION

This dissertation discusses a conceptual idea and testing gaps to improve SAW chemical detection by using Zeolite as the thin layer on quartz for an adsorption media. In review, there are many factors that can affect the sensitivity of the SAW sensor as shown in Figure 14 when considering the issues discussed in section 2.4 above. As mentioned earlier, a way of increasing sensitivity for detection is by increasing the operating frequency and/or sampling rate. The impact of increasing the operating frequency is best understood as discussed in [13-14]. Other researchers [7-8] are pursuing the sampling rates as well as flow control techniques to improve sensitivity of SAW chemical detection. Decreasing the film thickness and increasing selectivity can obtain further sensitivity improvements that Zeolite could be designed to utilize. Pure silica Zeolite (silicalite) films made by in-situ crystallization need to be developed because the hydrothermal synthesis procedure used is of concern to the bare SAW devices. The formation of porous zeolite films with surface patterns by convection-assisted dynamic self-assembly of zeolite nanoparticle suspensions shows promise [36].

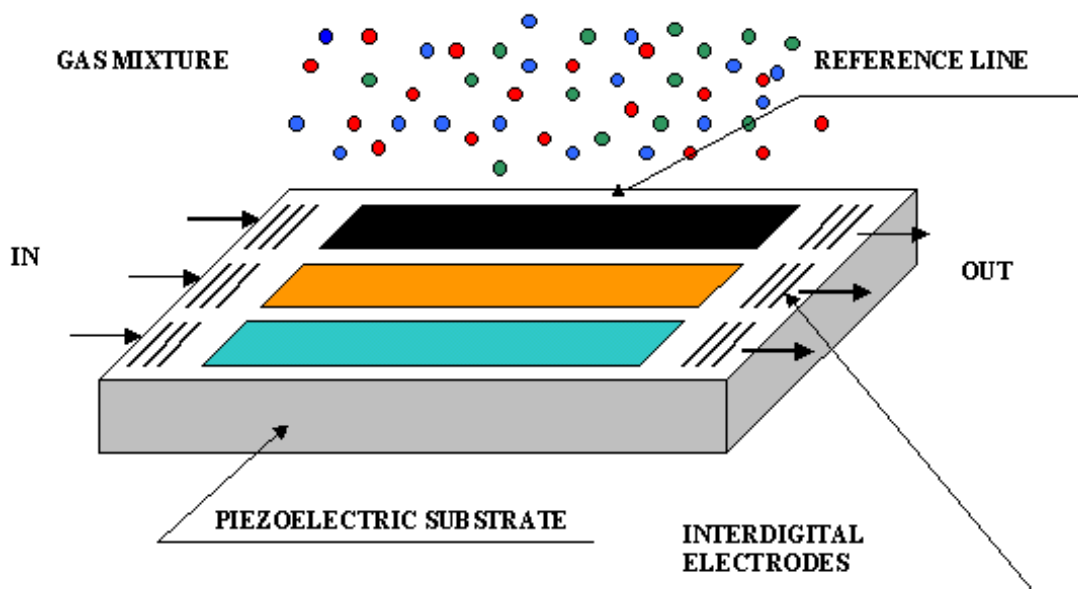


Figure 14. Drawing of SAW for Multiple Chemical Detection.

The frequency shift and insertion loss caused by the attachment of chemical agents on a thin polymer film coated on the surface of the SAW device is a mass driven response. Due to the thickness of the Zeolite layer, it is hypothesized that these nonlinear effects will be reduced vs. conventional polymer thin film coating.

The majority of published descriptions of SAW chemical sensors for organic vapors exploit polymeric films of varying thicknesses and selectivity to provide the required degree of sensitivity. Siloxane and fluropolymer are polymers being used for thin film in SAW sensors that can be tailored with the addition of a chemically selective monomer [7-8]. The distribution of the monomer is expected to be distributed throughout the thin film at the cross link points for the polymer, which can be verified with differential scanning calorimetry (DSC). It is believed that a new Zeolite manufacturing

can be developed as discussed in [36] with uniform porosity by embedding a particle in the process and chemically removing after the Zeolite has been crystallized.

Once the chemical vapor is transported to the surface of the coating on the SAW, the chemical adds to the mass of the coating via a transient non-uniform processes. Due to the abrupt change in the frequency response and insertion loss from the nonselective addition of mass, a chemical is detected by the resulting frequency change of several SAW's coated with different thin films. These frequencies are compared to known patterns or the abruptness of change to identify the presence of a chemical. Pattern recognition along with selectivity tailoring of the thin film has been the focus of researchers over the last 5 years [7-19]. An example of pattern recognition research is shown below from Andrew McGuill of Naval Research Laboratory in Figure 15.

To reverse the process, clean air is blown over the SAW, which results in another abrupt frequency shift. Since most SAW chemical identification uses pattern recognition based on a relative change in frequency, it is generally assumed that all of the chemicals have been removed from the coating when the frequency shifts back close to reference SAW level. As previously discussed, it can be realized that as the chemical diffuses out of the coating, the diffusion process slows down as discussed by Thompson and Stone in Reference [14] via $u_t = \alpha^2 u_{zz}$ and shown in Figure 4 above. Besides diffusion, an ionic chemical reaction may be created which requires Gibbs activation energy be overcome to remove the particle from the polymer. As well, the repeated cycling of a chemical into and out of the thin film may cause the coating to be physically aged which results in degradation versus time of the SAW detector as shown above in Figure 16 from

Reference [14]. Physical aging effects in polymers can be reversed by elevated temperature exposure above the polymer Tg for a specific time.

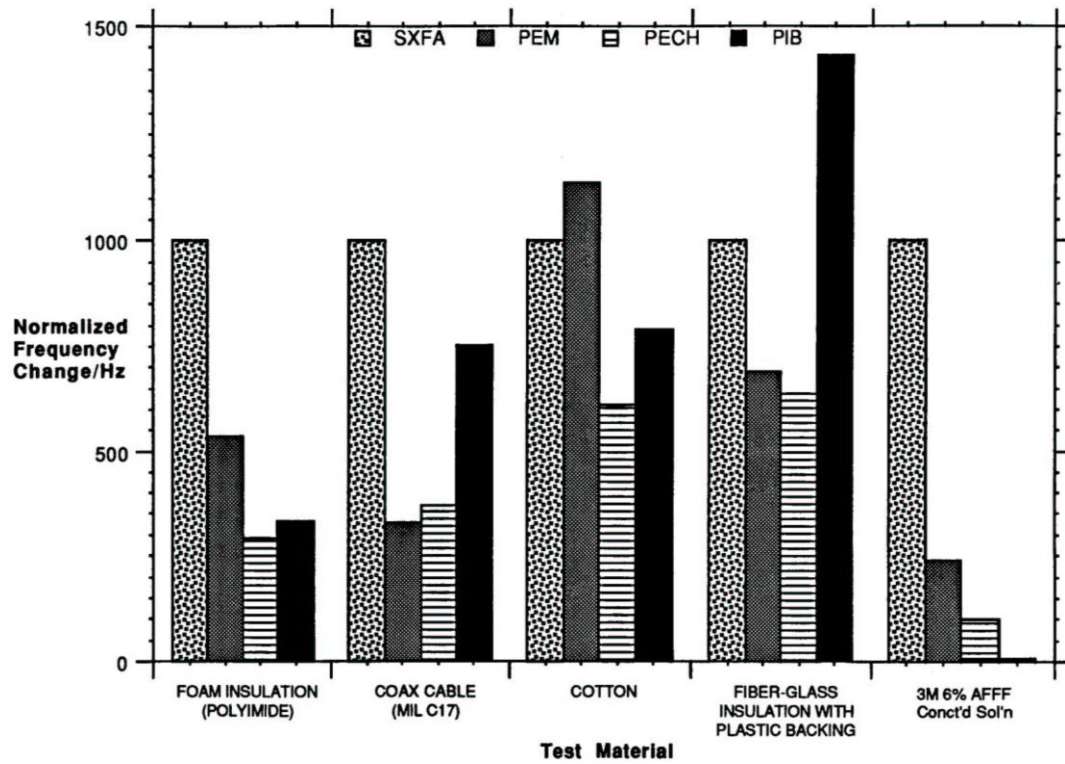


Figure 15. 4 SAW Array Pattern Responses Compared for 4 Heated Materials and AFFF a Possible Interferent.

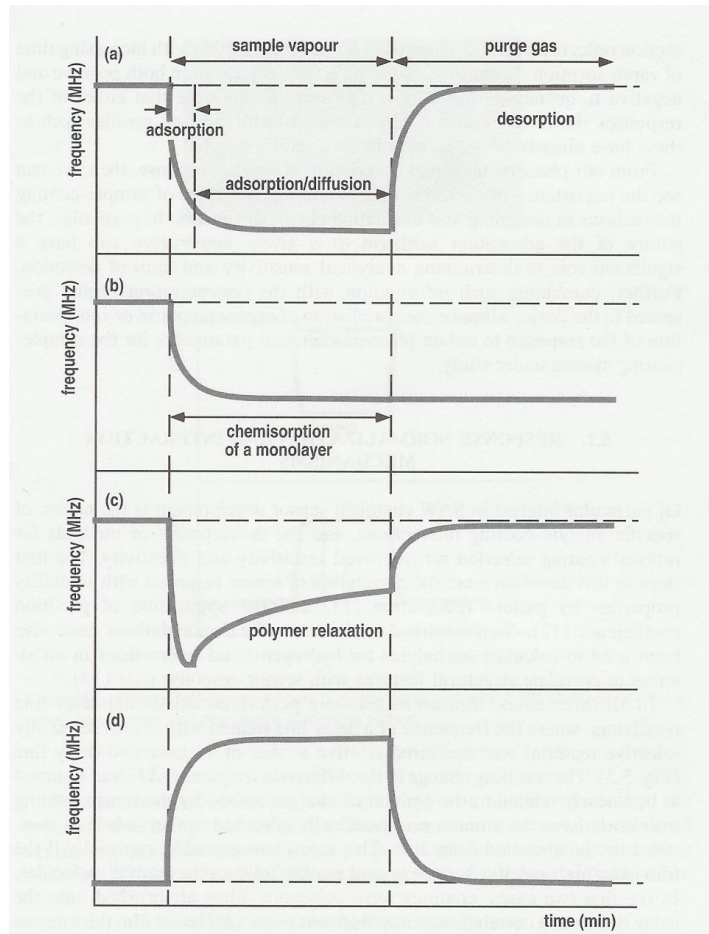


Figure 16. Possible response profiles of a coated SAW chemical sensor to sample vapor. (a) Ideal response, (b) irreversible chemisorption, (c) combined mass and viscoelastic effects, (d) large change in polymer properties for small mass absorbed.

However, it is being proposed that one of the SAWs be coated with Zeolite so that base line can be reestablished between detection events. Crystallized Zeolite does not degrade at temperatures below 500 °C. Hence, it is hypothesized that a temperature exposure of 550 °C could be used to clean the Zeolite coated SAW and provide a new baseline frequency for adjusting other SAWs hooked in parallel that have polymer coatings. The cleaning temperature of 550 °C would be used to burn any organics or

break any inorganic bonds established by detection events. As well, temperature further complicates the SAW detection process by shifting the elastic response as shown in Figure 17 from Reference [14]. It is also hypothesized that these and other moisture effects can be corrected for by getting an absolute baseline measurement from the Zeolite coated SAW.

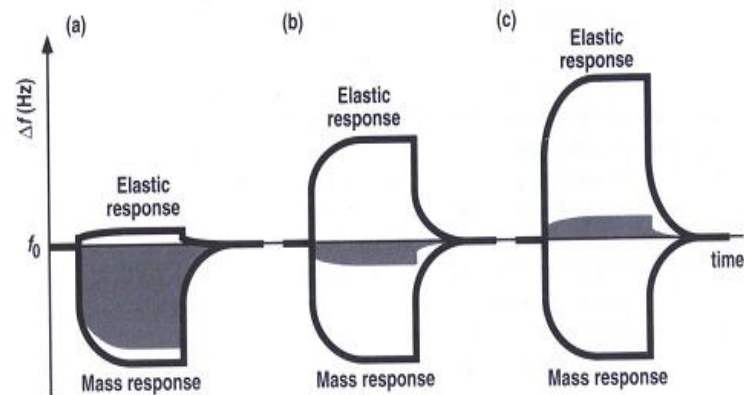


Figure 17. Effect of added mass and polymer property changes on SAW sensor response to vapor sorption. (a) $T_g, T_a > T_{\text{sensor}}$ and elastic effects are negligible. (b) $T_g < T_{\text{sensor}} < T_a$ Significant elastic effects. (c) As b, but elastic effects exceed change in mass and give a positive frequency response.

The largest producer of SAWs over the last decade has been for cell phones. Cell phones are a sophisticated radio that is strong enough to contact the cell tower in the center of a 10 mile grid. However, to keep the batteries small, a cell phone typically uses 0.6 to 3 watts of power and switches from cell to cell to maintain communication. From “How stuff Works” on the web, a typical cell phone can communicate in full duplex on 1,664 channels or more! To do all these channels, cell phones use three SAW chips which include a transmitter, receiver, and amplifier as shown in Figure 18. The chips

have aluminum as the thin film between the IDTs with a reference SAW used to eliminate environmental variation. The IDTs are put on with four step lift-off photolithographic process to generate aluminum IDTs on the surface of a quartz wafer as shown in Figure 5 [9]. Keeping accurate frequencies above 1 GHz was a challenge for quartz and required an intermediate SAW at about 500 MHz to clock the signal.

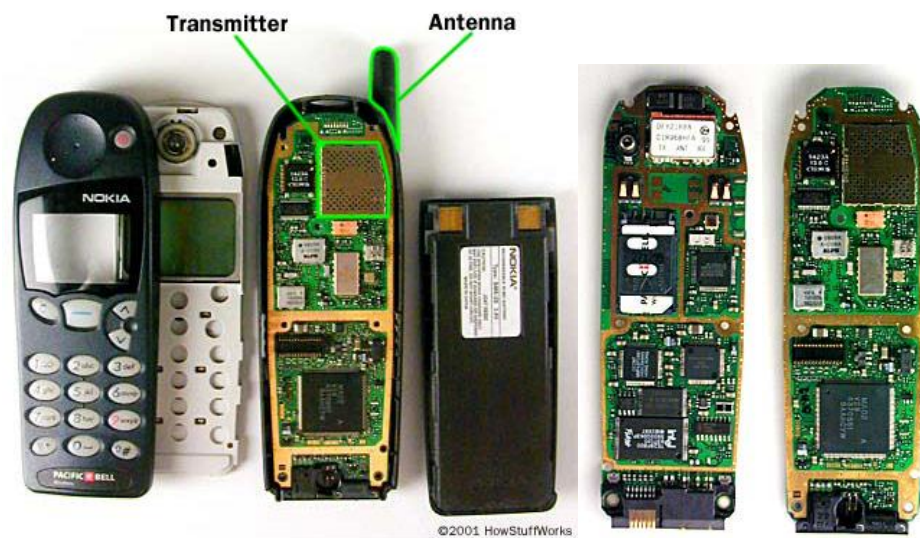


Figure 18. Nokia Phone.

As reproducibility has been addressed for quartz SAWs operating beyond 1 GHz, the commercial need for 500 MHz SAWs has been reduced. This lead to an opportunity with Vectron Company in the form of a None Disclosure Agreement (NDA) to develop SAW sensors that operates in that frequency range. The frequency spectrum from Vectron is represented in Figure 19. Vectron's SAW application product list relative to frequency is shown in Figure 20 to understand market drivers for SAW technology. The SAW product list is quite extensive and limited in Figure 8 to a few relevant applications

relative to Figure 18 for understanding the issue of spectrum. Other technology issues currently occurring in cell phone design focus on reducing all the radio functions to one SAW such as Silicon Laboratories Si4206 Aero I GSM/GPRS transceiver.

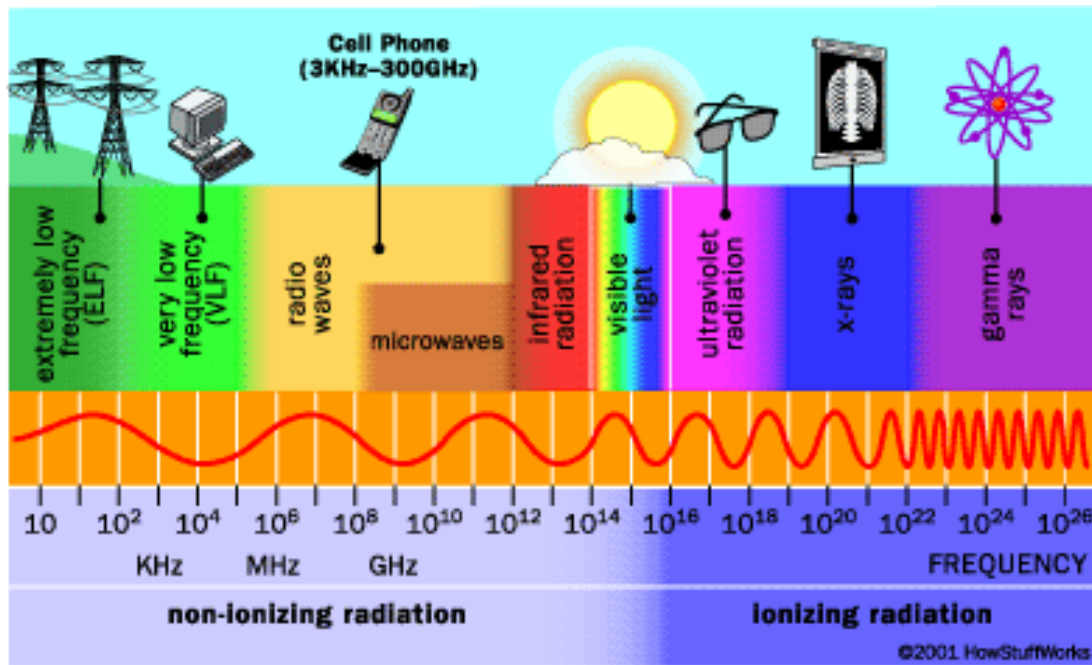


Figure 19. Frequency Use Layout.

<u>Application</u>	P/N	Center Frequency	3 dB	Pass Band Ripple	Insertion Loss	Group Delay Ripple	Package
Routers, LMDS	TFH36A	36.125 MHz	8.0 MHz	0.55dB	27.5dB	70ns	24 mm x 9 mm LCC
Wireless Communication	TFS36C	36.0 MHz	1.50 MHz *	1.0dB	36.0dB	120ns	35 mm x 20 mm DIP
Mobile Communication Systems, GSM,DCS,PCS	TFS45A	45.5 MHz	340 kHz	1.5 dB	6.5dB	300nS	21 mm
Measuring Device for GSM,PCS Transmitter	TFS52	52.0 MHz	270 kHz	0.5 dB	15.0dB		35 mm x 12 mm DIP
Digital Radio Systems	TFS70H	70.0 MHz	0.25 MHz to 40 MHz		28.0dB		22.1mm x 12.62mm 6/14 pin/DIP or 27.18mm x

Bandselective repeater, Cellular	TFS70S	70.0 MHz	10.8 MHz	1.0dB	24.0dB	200ns	35 mm x 20 mm DIP
DECT, Bluetooth	TFS111	111.0 MHz	900kHz		14,0dB	300ns	13 mm x 6 mm LCC
GPS	TFS135C	135,42 MHz	2,5 MHz		15,5dB	80ns	7 mm x 5 mm LCC
Digital Repeater, Radio, LMDS	TFS140	140.0 MHz	6.00 MHz	1,0dB	24.0dB	80ns	22 mm x 13 mm DIP
Low power radio	TFS433C	433.92 MHz	4.0 MHz		3.5dB		7 mm x 5 mm LCC
Keyless entry	TFS433E	433.92 MHz	240kHz **		5.0dB		9 mm x 5 mm LCC
GSM,DCS,PCS	TFS440	440.0 MHz	200 kHz		6.5dB	2.0μs	5 mm x 5 mm LCC
Low power radio	TFS460B	460.0 MHz	20.0 MHz *	2dB	5.0dB		7 mm x 5 mm LCC
Wireless Communication	TFS462	462.0 MHz	4.0 MHz		4.5dB		F11
Pager RF	TFS469	469.99 MHz	280kHz **		5.0dB		9 mm x 5 mm LCC
Wireless Communication	TFS600	600.0 MHz	32 kHz *		6.5dB		7 mm x 5 mm LCC
VCSO	TFS622	622.105 MHz	140 kHz *		6.0dB	600ns	7 mm x 5 mm LCC
Stable Oscillators	TFR434A	434.0 MHz			1.8dB		5 mm x 5 mm LCC
** typical value * 1 dB Bandwidth							

Figure 20. Partial Vectron SAW product list.

A single notch filter sample for testing development was supplied by Vectron as part of the relationship as shown below. Requests to purchase less than 20 SAWs resulted in a quantity limit. As defined by the RF sensor industry, a limited quantity is defined as sensors available on the warehouse shelves. Price and demand are tied together by economics resulting in the price to escalate for limited quantities since the sample SAW from Vectron was out of production as well. Although frustrating, this

setback lead to looking at other SAW technologies and discovering a dissertation as shown in [42]. A phone conversation discussing the availability issues with Lane Manoosingh resulted in him recommending RF Monolithics (RFM) for small quantity SAWs. As a result of wanting to study different frequencies, technology, test procedures and price, a Code Division Multiple Access (CDMA) 9 pin surface acoustic wave chip from RFM was initially purchased in a quantity of 50. It was later understood that the chip was utilized by Verizon for cell phones.

The use of a conducting or insulating thin film leads to many design challenges that have been addressed by Thompson & Stone in Reference [14] and shown in Figure 21 below. Most of these results came from empirical data that needs to be evaluated for a Zeolite thin film. The Zeolite absorbing film can be put on using a spin coat or deposition technique to generate a thin coating.

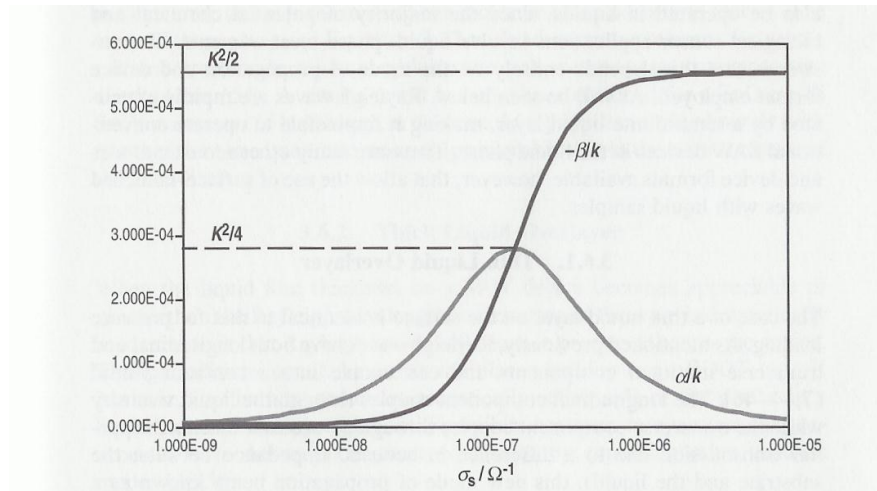


Figure 21. Plot of the attenuation and fractional velocity change as a function of surface sheet conductivity according to Equations 3.10 and 3.11. Calculation for $C_s = 0.5 \text{ pF cm}^{-1}$, $K^2 = 0.0011$ and $\lambda = 60 \text{ }\mu\text{m}$.

[14]

The dynamic test set ups will entail a spectrum analyzer hooked to an oscilloscope to switch between time and frequency domain with a signal generator or the static test setup will entail a network analyzer hooked as idealized in Figure 22. These devices will be monitoring the SAW frequency response when exposed to a concentration of gas as shown in Figure 23.

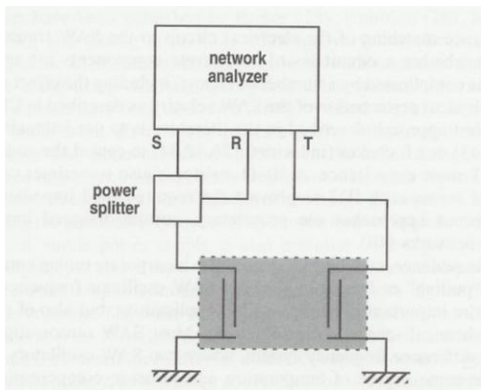


Figure 22. Use of a spectrum-network analyzer for the measurement of SAW delay-line properties.

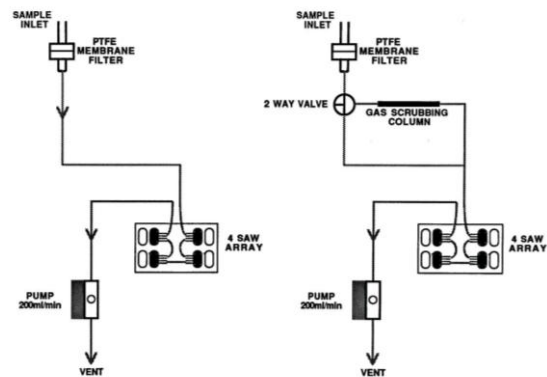


Figure 23. Ambient gas Sampling Systems.

3.1 Phase I Testing

In the pursuit of dynamic measurements, goals were developed using a combination of voltage and acoustic equipment to evaluate the SAW device response. Real time monitoring was to include a Spectrum Analyzer (SA) connected to the SAW device to generate a frequency response. A network analyzer was to be utilized to determine the test setup impedance, voltage and delay. Finally, the plan was to implement gas diffusion into a film with the frequency response monitored real time. Then, the plan was that the SAW would be cleaned to show it can be cleaned without degradation effects. However, this effort was shifted to a future goal.

Three operating frequencies were chosen for the initial study devices. RF Monolithics (RFM), Vectron and Microsensor Systems (MS) produces pin mount SAWs at the 70, 155.2, and 245 MHz central frequency levels. The RFM BP1042 SAW was picked as a low cost keyless entry system. Vectron's AT&T SAW was picked as an intermediate operating frequency SAW. MS commercially produces gas detection systems; some of which are based on this SAW. The hermetic covers were removed and a picture taken is shown below in Figure 24.

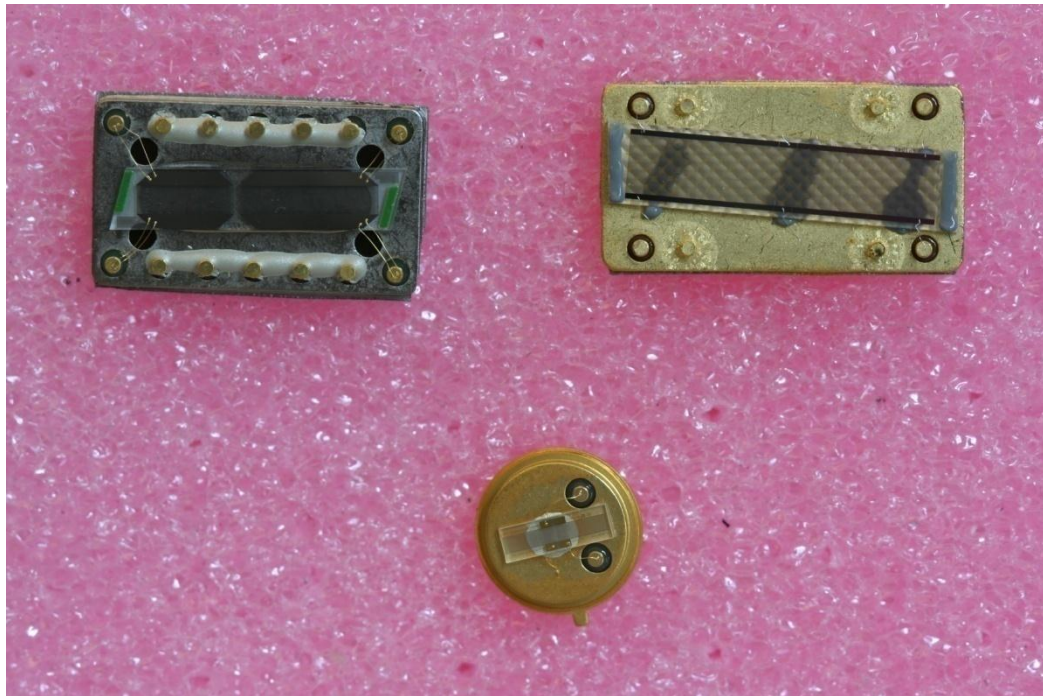


Figure 24. Photos of Vectron SAW (top left), RFM SAW (top right), and MS SAW (bottom) without covers.

Although obvious at a later time, the initial testing on bare SAWs was performed with alligator and plunger clips attached to SAW pins as shown in Figure 25. The measurement accuracy was identified as low due to electrical variability caused by the SAW pins acting as antennas. This was addressed by designing a PC board with integrated plug adaptors and properly shielded cabling. Faults in initial PC board design were discovered when all SAW types did not operate as expected. The second board design with appropriate adaptors is shown in Figure 26. SMA connectors provide the grounded input and output for the SAW mounted between SMA RF connectors on the printed circuit board (PCB) as shown in Figure 27 & 28. Although noise was greatly suppressed, further techniques with software and/or hardware filtering were required to completely address the noise issue in these experiments which will be discussed later.

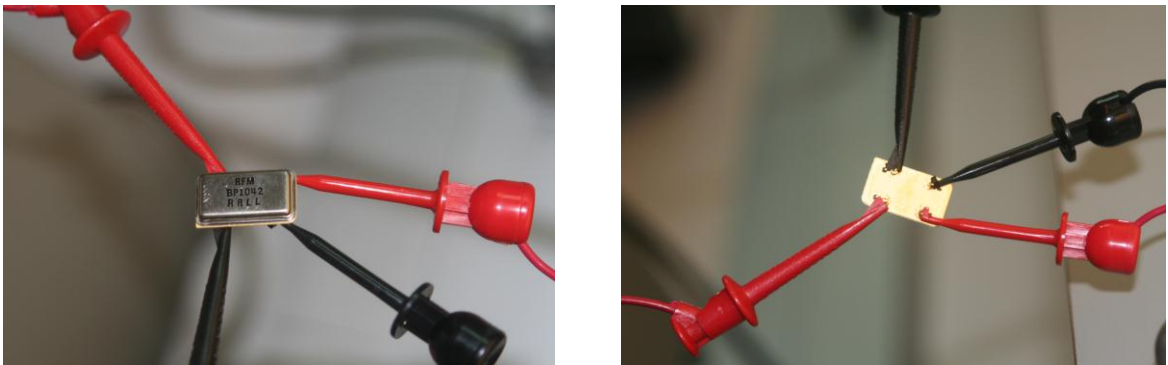


Figure 25. Initial Plunger Clip Experimental Test Setup.

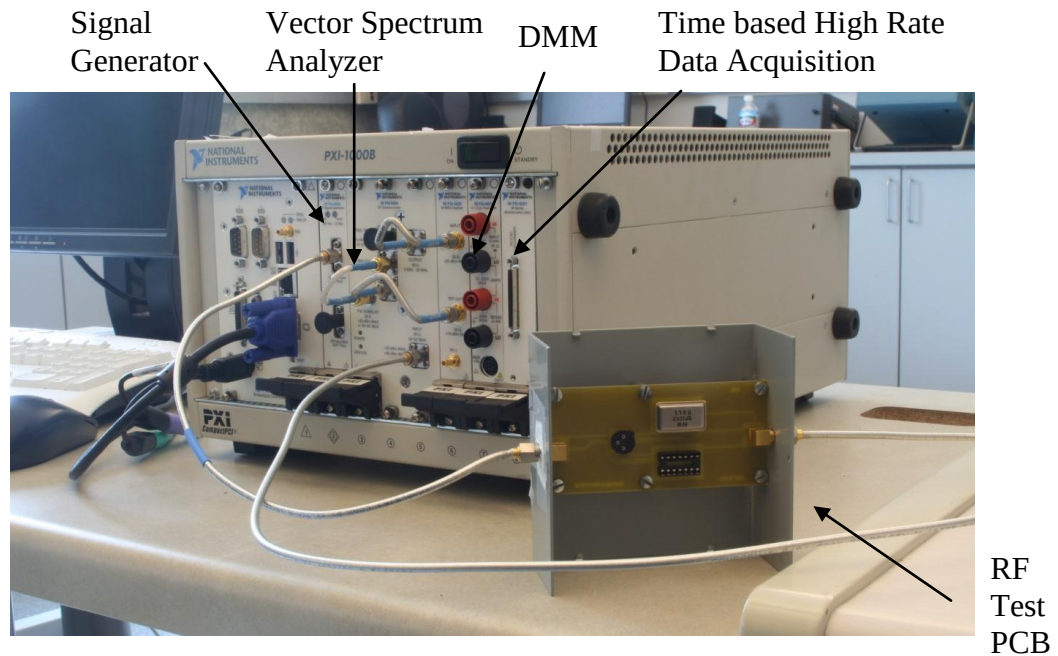


Figure 26. Initial Spectrum Analyzer Experimental Test Setup.

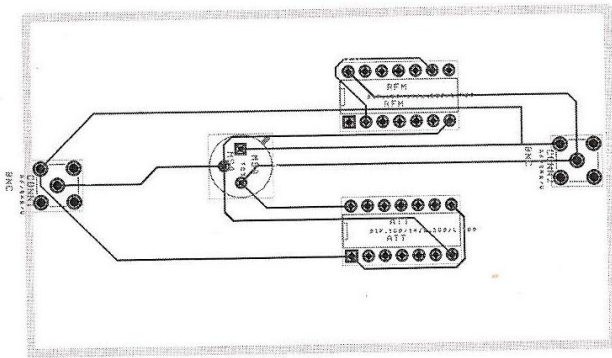


Figure 27. Scanned Image of the PCB Design.

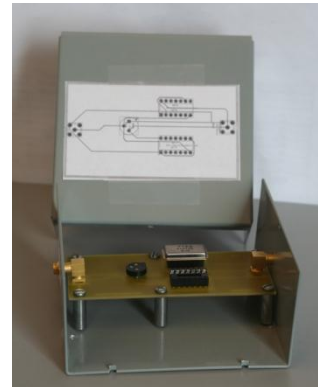


Figure 28. Image of the PCB. Design and Shielding Box.

Initially, all of the SAWs were evaluated with a microscope as shown in Figure 29, 31 & 33 and for response with the SA setup shown above in Figure 26. The SA setup results are shown in Figure 30, 32 & 34. Since more of the 154 MHz SAWs could not be

obtained, the 70 MHz RFM SAWs were selected for future testing to develop the test technique. This research was not focused on developing SAWs, but rather understanding how frequency and design changes might impact gas detection.

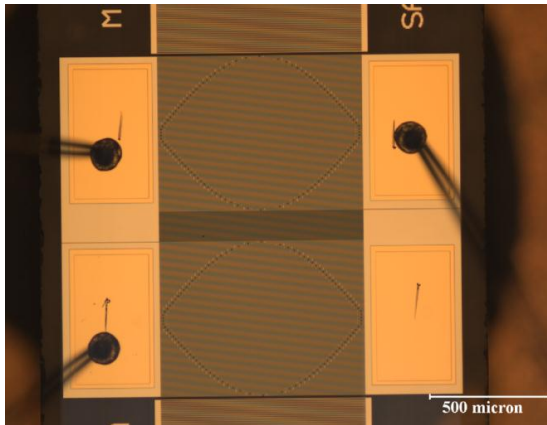


Figure 28. MS SAW
(248 MHz) = \$300/each.

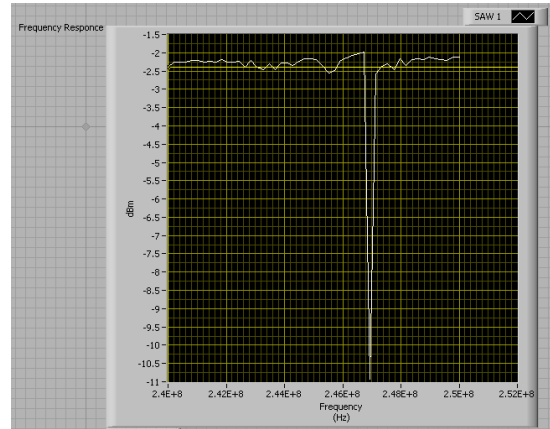


Figure 29. MS SAW
(248 MHz) Frequency Response.

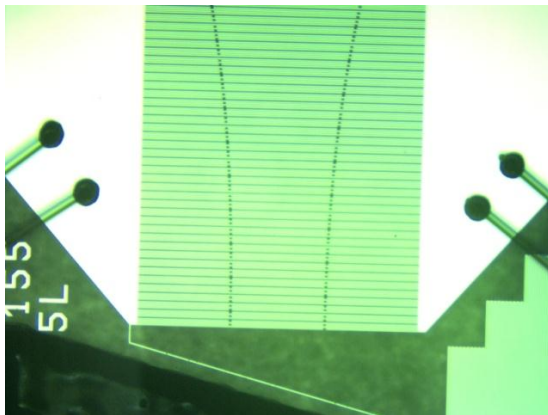


Figure 30. Vectron ATT SAW
(154 MHz) sample.

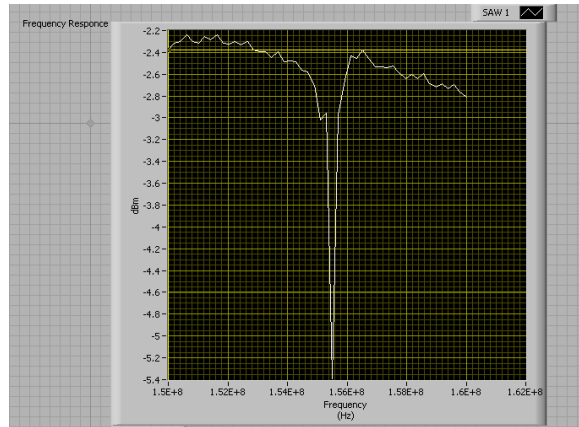


Figure 31. Vectron SAW
(154 MHz) Frequency Response.

It is surmised that the multiple peak response of Figure 33 below may allow for signature analysis that will allow for improved sensitivity in frequency shift. As well, the variations may allow for identification.

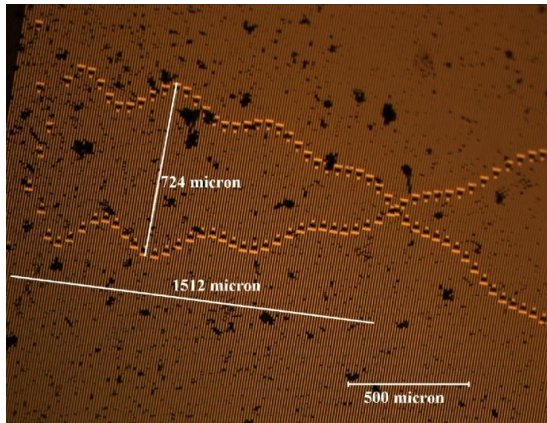


Figure 32. RFM SAW
(70 MHz) = \$3/each.

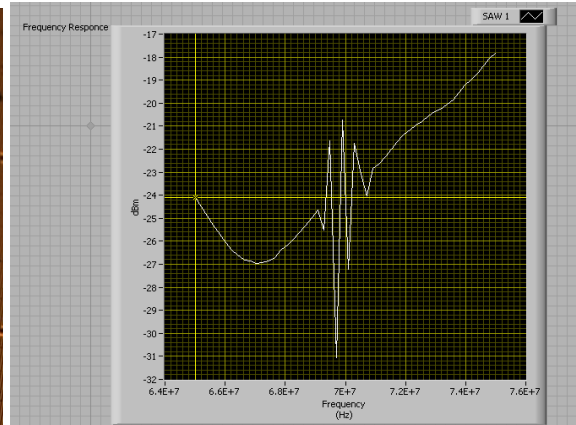


Figure 33. RFM SAW
(70 MHz) Frequency Response.

Although not shown here, the SA SAW testing results generated more questions than answers due to response variability. The main purpose of the experimental research was to develop quantitative results to better understand the issues related to SAW chemical sensor calibration. So, the SA testing was postponed until after the frequency shift static measurement method was developed and proven. The dynamic measurement with SA and gas delivery, as shown in Figure 35, requires more circuitry and a lot more complexity to obtain a qualitative measurement with gas adsorption.

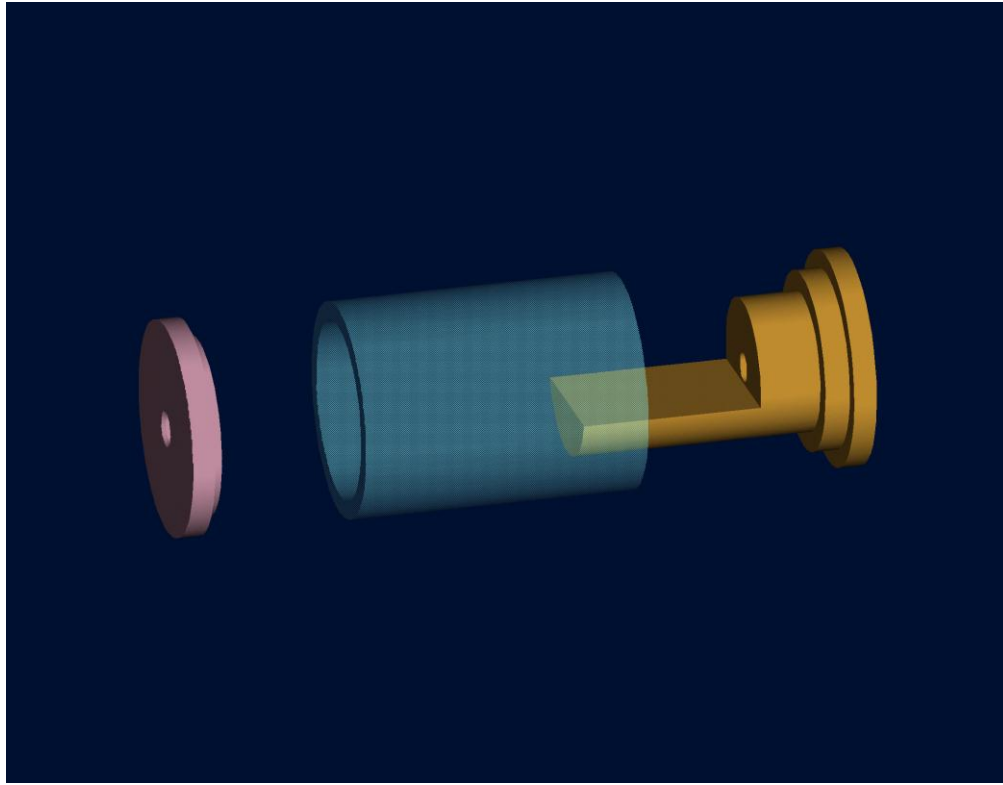


Figure 35. Gas delivery apparatus concept with SAW inserted into adaptor that is integrated into PCB on shelf and leads exiting through connecting plugs.

3.2 Phase II Testing

Hence, a static measurement setup was developed as shown below in Figure 36. The NA was utilized to understand sources of variability in the testing process. Initial testing focused on observing the frequency shift.



Figure 36. Network Analyzer Experimental Test Setup.

So, a RFM SAW was tested to understand the response as shown below in Figure 37.

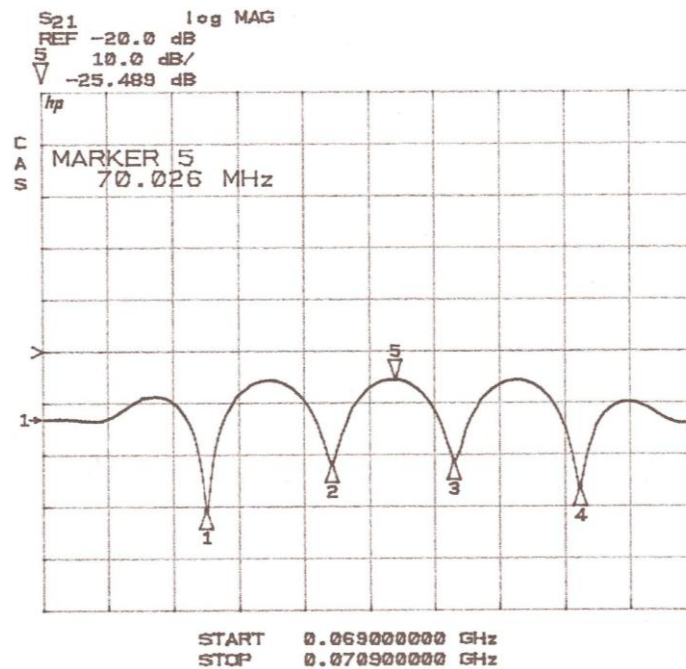


Figure 37. Hermetically sealed RFM SAW2 Frequency vs. Attenuation.

After SAWs have hermetic seals milled off, they are tested to assure no damage has been induced from the process. Damage results in a flat line due to continuity of electrical connection being broken. Below is initial testing before & after covers were removed as shown in Figure 38 & 39. This demonstrated that the NA could realize the frequency shift statically. The frequency and amplitude values for markers in Figure 37-39 are shown in Table 1 below.

Marker 1	69.475 MHz	-46.814 dB
Marker 2	69.836 MHz	-40.301 dB
Marker 3	70.197 MHz	-39.963 dB
Marker 4	70.558 MHz	-45.42dB
Marker 5 (max)	70.0165 MHz	-25.863 dB

Table 1. RFM SAW2 Response Markers.

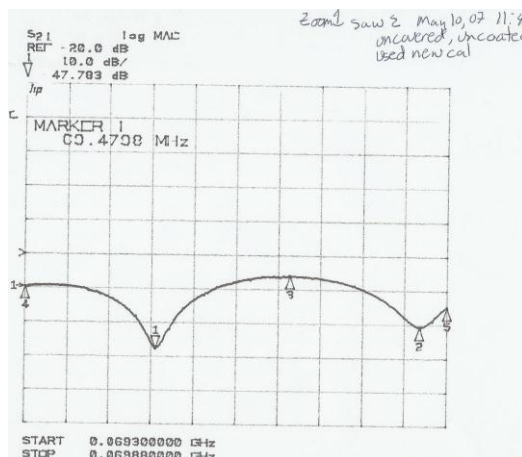


Figure 38. Zoom Covered RFM SAW2 Frequency vs. Attenuation.

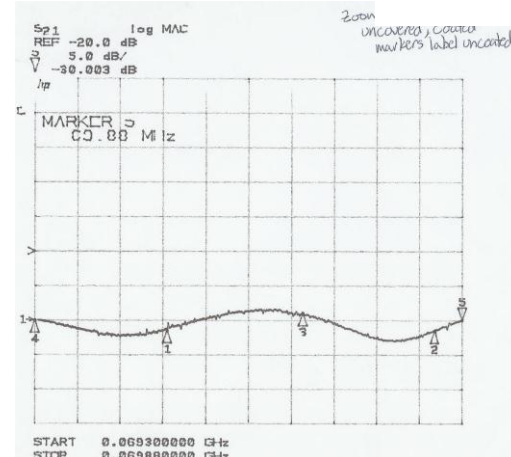


Figure 39. Zoom Uncovered RFM SAW2 Frequency vs. Attenuation.

The next step in testing was to apply a thin film by spin coating. Spin coating was utilized, because current manufacturers are applying polymer films with this technique. Since polymer resins are more complicated, water glass was utilized to understand the process and repeatability. It was believed that the water glass film would not have a transient response if environmental variable variations were kept to a minimum by storing under vacuum.

The SAW is held in a pin mount socket assembly as shown in Figure 40. Then the films are deposited on the SAW surface with a spin coater (WS-400A-6NPP LITE by Laurell) shown in Figure 41. The deposition process includes placing the SAW in the chuck with the vacuum on and the chuck not rotating. Then, drops of waterglass were placed on the SAW and the lid closed with the spin coater revolving at 6000 rpm for 2 minutes. Currently, no cross checks of film thickness has been confirmed but it is assumed that the SAW is uniformly coated based on prior thickness calibrations of waterglass film and rotational speeds.

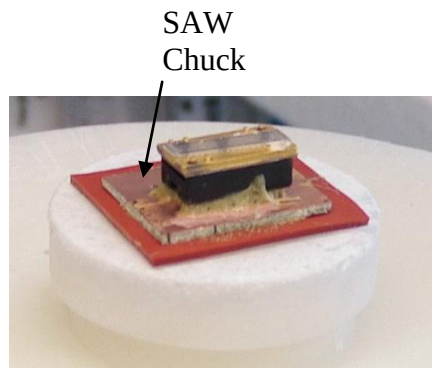


Figure 40. Vacuum SAW Chuck.



Figure 41. Rotational Spin Coater.

The films deposited with the spin coating technique are highly variable due to distribution of source liquid material. The variability can be observed in Figure 42 where a drop of water glass was spun coat. By utilizing a prior sputter coating of the SAW, it is believed that a better spin coating result could have been developed.

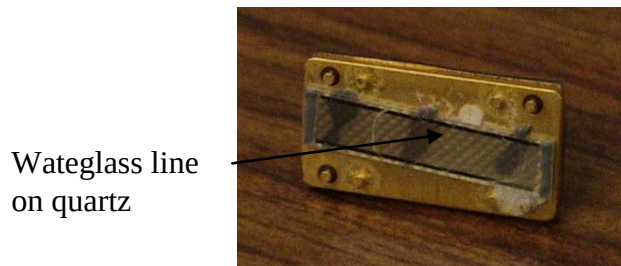


Figure 42. *Variability Spin Coating.*

Initially, an attempt was made to characterize the film thickness generated by different spin speeds of water glass on quartz. However, due to measurement difficulties it was decided to utilize the water glass on glass calibration results as a first estimate of film thickness as shown in Table 2. The thickness was measured with a surface profiler in the UCR clean room. An initial result shows that the pure water glass at the highest revolution speed of 6000 RPM for 2 minutes duration generated too much damping at 70 MHz for the frequency shift to be detected with a thickness of 3.3 μm as shown in Figure 43.

RPM	TIME (min)	THICKNESS(μm)
6000	2	3.3
6000	3	3.5

Table 2. Film thickness on glass slide using Spin pure waterglass.

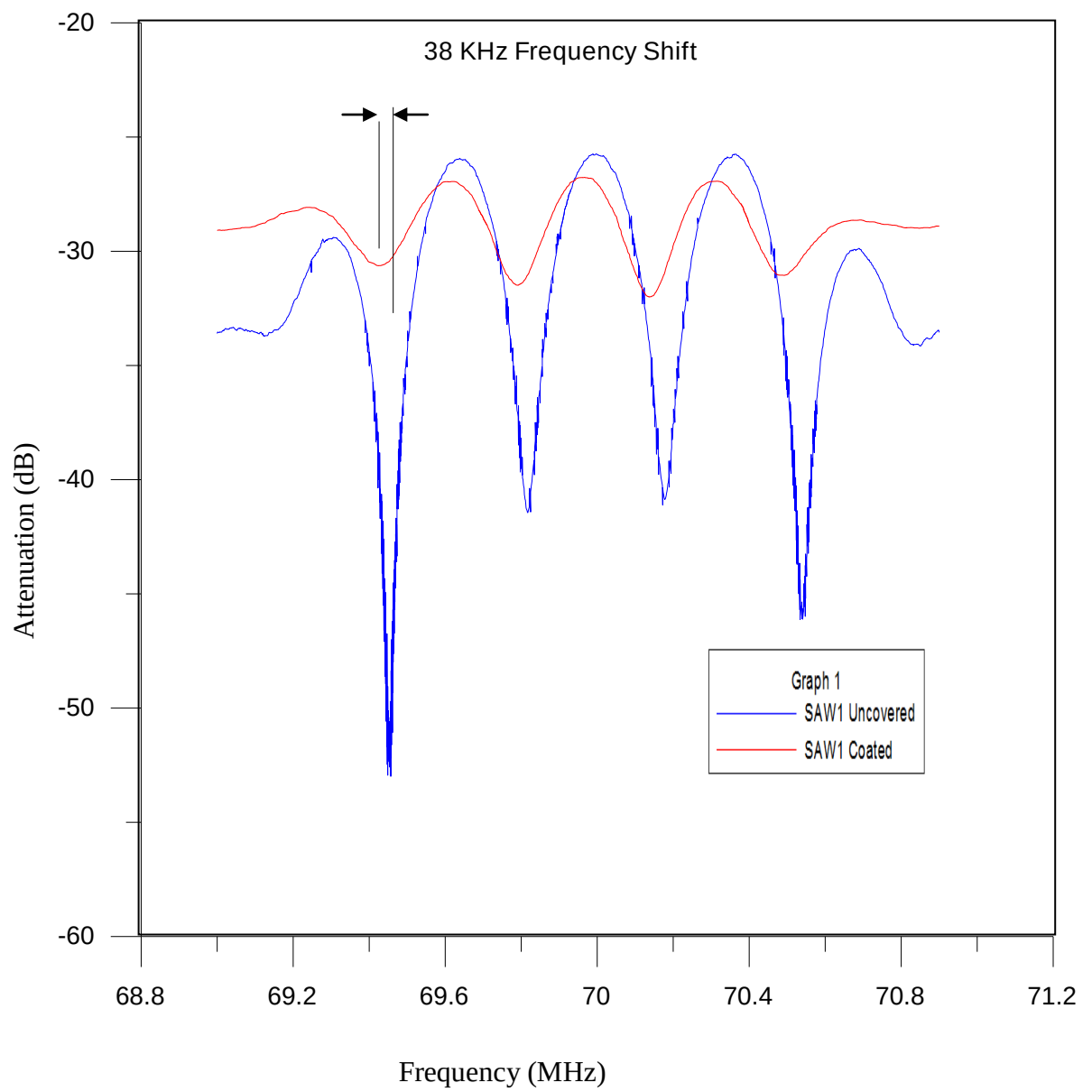


Figure 43. RFM SAW1 Frequency vs. Amplitude Shift Results.

Only one SAW and thickness was applied and tested. The estimated frequency shift was 38 KHz as shown in Figure 43. Due to the film variability and damping, this technique was abandoned from lack of control. An approach for overcoming the poor surface spreading with spin coating was identified to sputter a coating of SiO₂ to develop a smooth surface. The irregular surface caused by the difference in IDT metal height and piezoelectric surface would require a test matrix to optimize thinness of sputtered film.

3.3 Phase III Testing

A sputtering technique was developed for applying SiO₂ films to all other SAWs for testing. The sputtering system is an ATC ORION 5 UHV by AJA International Inc as shown in Figure 44. The accuracy and quality of spin coating were reasons for switching to sputter coating. Along with uniformity, selectivity, reuse and repeatability of sputter coating film across SAW were reasons for switching from Spin Coating. A calibrated deposition rate was followed for SiO₂ = 0.068 Angstrom/s.

In order to place the SAW into the ultra high vacuum chamber for SiO₂ film deposition, the SAW was attached to a styrofoam using a double sided tape. The sample was then transferred to the main chamber using a load lock. The specimen holder and vacuum insert holder is shown in Figure 45 and 46.



Figure 44. The ATC ORION 5 UHV Sputtering System used for SiO₂ film deposition.



Figure 45. Vacuum insert holder.

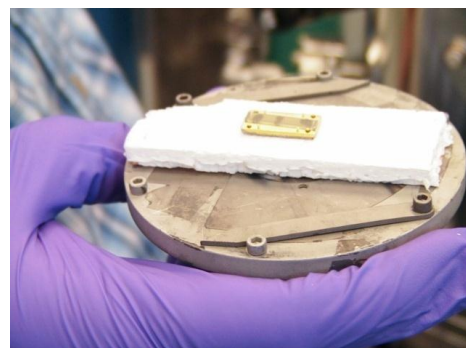


Figure 46. Specimen Holder.

Initially, very thin films were produced to observe the frequency shift. Mishandling the bare SAWs resulted in a lot of flat lines. Further, testing irregularities resulted in a lot of noise as shown in Figure 47. The variability was initially addressed by using an aluminum cover as shown in Figure 48.

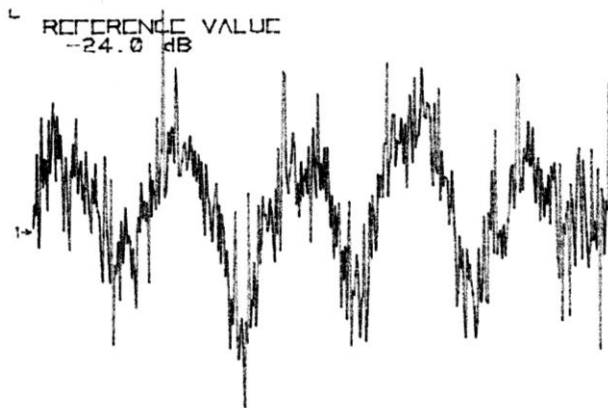


Figure 47. Noise Results.



Figure 48. Aluminum over cover.

However, the flat line was found to be caused by the box developing a charge. This problem was solved by grounding the box through a power conditioner as shown below in Figure 49 and 50. The NA test result after the grounding change is shown in Figure 51.

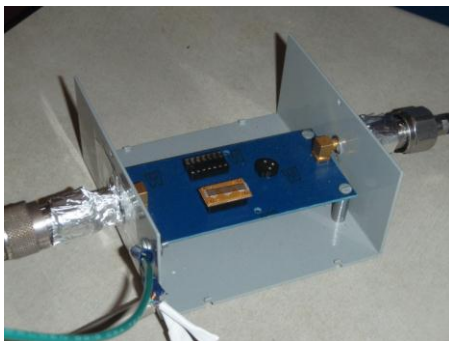


Figure 49. Vacuum insert holder.



Figure 50. Specimen Holder.

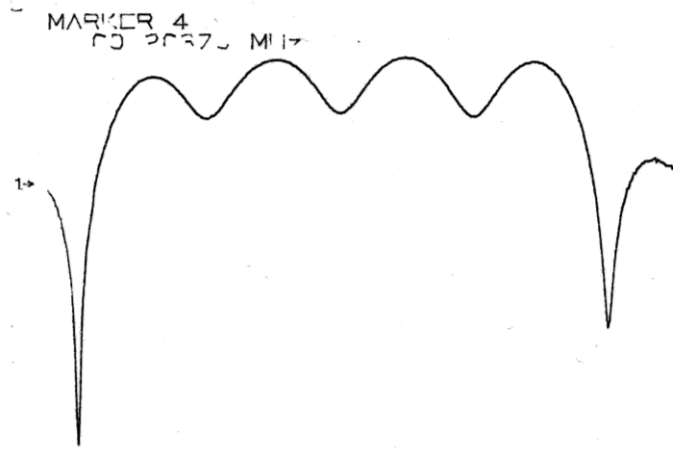


Figure 51. *Grounded Device Under Test (DUT) Box results
Which is also the response shape for a sealed SAW.*

The specific NA procedure is shown below which was logged in a log book for error checking.

Detailed Network Analyzer Initial Test Procedure

- a) calibrate
- b) note date and time
- c) generate results, place and document markers
- d) plot results with grid on and off
- e) keep track of direction
- f) scan plots in and convert to digital
- g) check scaled results to documented marker numbers

Sauerbrey made the first quantitative investigation with a vacuum deposited thin film and derived a linear decrease in frequency change with added mass [41]. The testing generated results are shown in Table 3 which generated the expected linear result shown in Figure 52. The sputter time between each frequency measurement test date was 58820 s. The mass was calculated using a coverage area for the SAW of 0.113 cm^2 and a density of quartz of 2.203 g/cm^3 . However, the unexpected frequency response changes in shape as shown in Figure 53-55 created further research.

		<u>5 29 to 6 03</u>	<u>6 04 08</u>	<u>6 05 08</u>	<u>6 06 08</u>	<u>6 07 08</u>	<u>6 18 08</u>	<u>6 19 08</u>
		frequency shift	frequency shift	frequency shift	frequency shift	frequency shift	frequency shift	frequency shift
		kHz	kHz	kHz	kHz	kHz	kHz	kHz
		423.5	476.375	388.875	348.75	348.75	258.75	0
	means Δf	437.375	471.25	387.625	360	348.75	270	0
		394.875	886.375	403.75	360	337.5	258.75	0
		416.875	938.125	413.75	360	348.75	270	0
		435.875	481.5	49.5	371.25	337.5	258.75	0
		438.125	539	1384.125	343.125	353.375	269.375	0
		427.125	476.375	389.25	343.125	358.5	274.5	0
		394.875	881.25	394.375	348.25	358.5	274.5	0
		427.125	953.5	404.625	353.375	363.625	284.75	0
		418.375	481.5	40.75	348.25	363.625	274.5	0
		423.352		384.393	344.525	353.797	271.684	0
		412.687		376.09	342.572	356.328	264.292	0
		423.327	455.201	376.649	342.684	356.075	264.292	0
		572.811	1470.567	377.921	336.212	354.05	264.761	0
		302.651	419.162	403.633	450.467	350	260	0
			462.057					

Table 3. Frequency Shift Data.

Utilizing Sauerbrey Equation 4 shown below.

$$\Delta f = \frac{-2\Delta m f_0^2}{A\sqrt{\rho_q u_q}} = \frac{2f_0^2}{A\sqrt{\rho_q u_q}} \Delta m \approx -360 \text{ kHz} \quad (4)$$

f_0 – Resonant frequency (Hz)

Δf – Frequency change (Hz)

Δm – Mass change (g) = $1 \times 10^{-5} \text{ g}$

A - Piezoelectrically active crystal area (Area between electrodes, cm^2) = $\frac{0.113}{4} * \text{cm}^2$

ρ_q – Density of quartz (2.648 g/cm^3)

u_q – Shear modulus of quartz for AT-cut crystal ($u_q = 2.947 \times 10^{11} \text{ g / cms}^2$)

Sauerbrey's Equation is greatly effected by active crystal area which supports the need for a approximation analytical technique that utilizes the admittance pattern of the SAW.

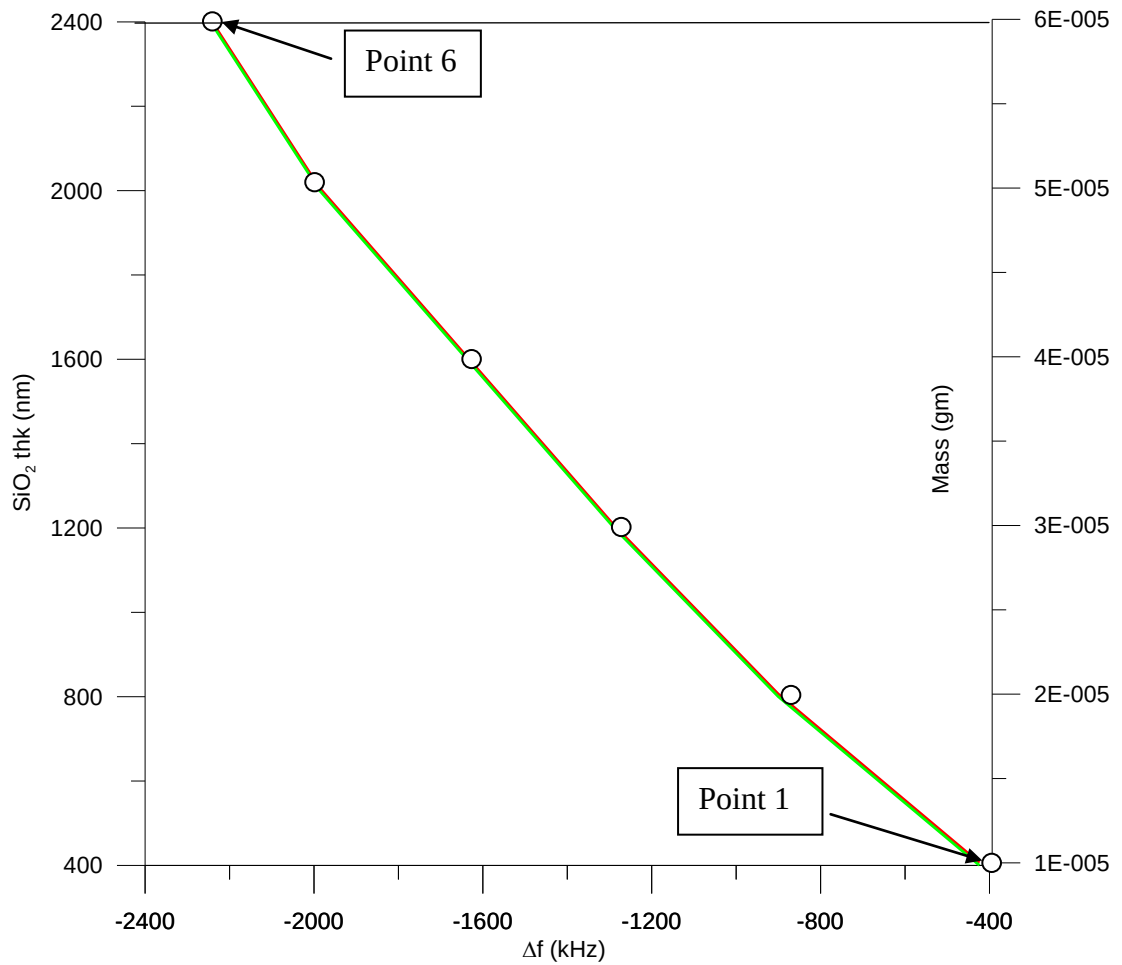


Figure 52. Frequency Shift Results.

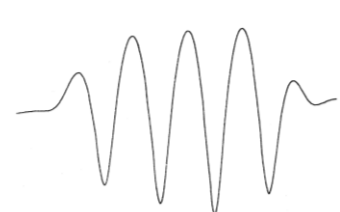
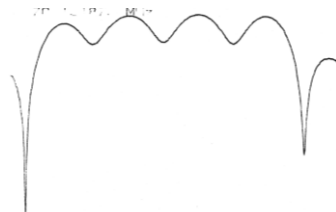


Figure 53. Point 1 Response. **Figure 54.** Point 3 Response. **Figure 55.** Point 6 Response.

Most knowledge of SAW performance as a chemical sensor is based on experimentally derived equations or models. Initial effort was focused on understanding frequency shift issues. Due to a large number of SAW failures (50+), a search for a reuse

capability using tapes to obtain more data points for test results was initiated. The only solution was to sputter a thin coating of SiO_2 to not ground between conductor lines after covering the wires with polyurethane as illustrated in Figure 56. The pattern shown in Figure 56 is created by the gap between the positive and negative IDTs as shown in Figure 57 for the RFM SAW. The IDT's width was determined to be $5\text{ }\mu\text{m}$ for the RFM SAW used in these tests. Microscope pictures were difficult due to uneven pin lengths and focus at $1\text{ }\mu\text{m}$ scale due to light interference as shown in Figure 58. Further complexity in testing was realized as the visually obvious manufacturing differences in RFM SAWs are shown in Figure 59. The manufacturing differences were not evaluated but separated to not effect results.

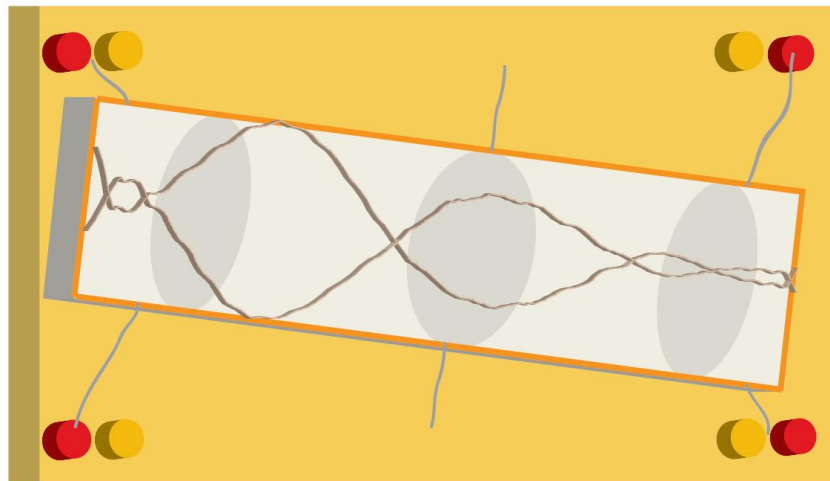


Figure 56. *Illustration of RFM Test SAW.*



Figure 57. *Optical microscope photos of RFM IDT Pattern.*



Figure 58 *RFM IDT Size.*

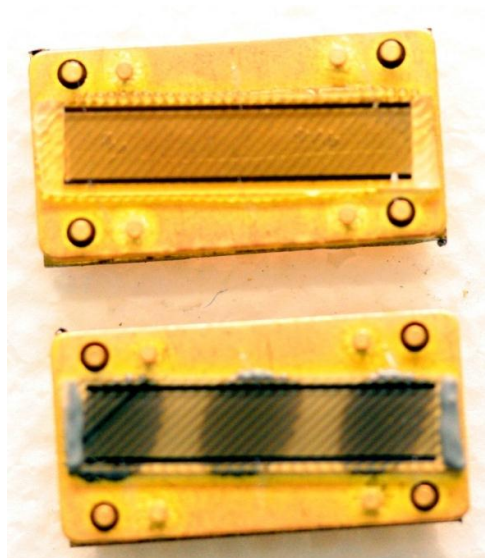


Figure 59 *RFM Visible Manufacturing Differences.*

4. RESULTS AND DISCUSSIONS

4.1 Phase IV Testing

The test method was developed to observe the frequency shift and address measurement repeatability. After the manual scanning technique was developed to digitize the data, a new question arose were the results varied in a patterned way as shown above in Figure 56 to 58. This tuning procedure shown below was to address repeatability in SAW response.

1. Match hermetically sealed SAW response through physical adjustments in placement and rotation
2. Immobilize placement and replace with test SAW

Overall repeatability of each test is shown in Figure 60 within the accuracy of overall testing at ± 30 kHz. The sensitivity of tuning in amplitude and frequency is more easily observed in Figure 61 with worst case ± 87 kHz inaccuracy induced by tuning sensitivity. This inaccuracy caused a change in the tuning process that included matching frequency and amplitude. The implementation of this step removed the test induced responses. The need to implement this procedure just demonstrates the sensitive of the SAW technology.

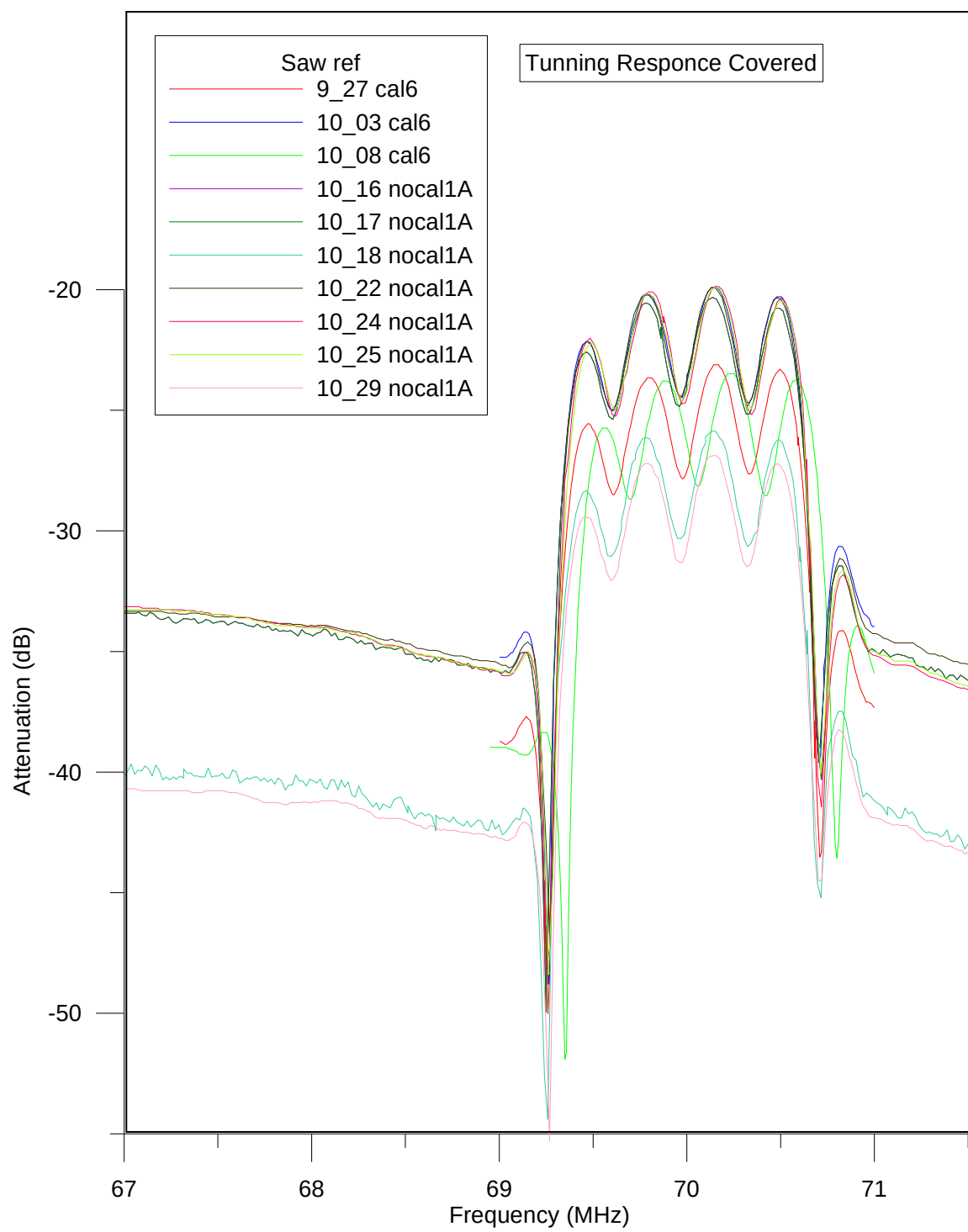


Figure 60. Repeatability Tuning Plot.

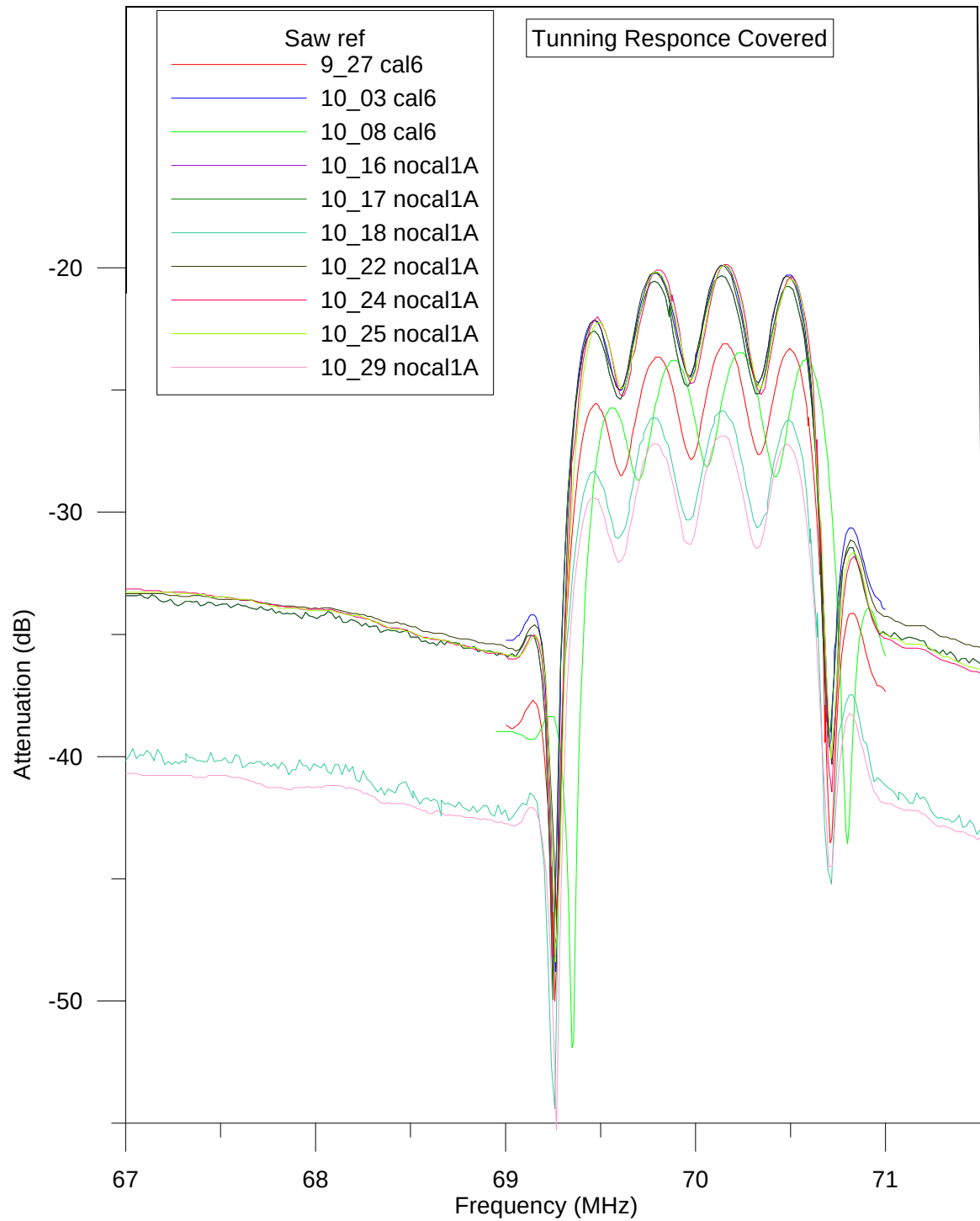


Figure 61. Worst Case Inaccuracy Tuning Plot.

Utilizing the tuning for two RFM SAWs (62 & 64) tests resulted in data from covered, uncovered and 10 separate 400 nm thick SiO₂ layers for mass change. Hence, the testing ranges from no mass to 4 μm thick to span the spin coated water glass. Understanding accuracy is driven by span and number of points for each curve plotted from the NA. Then the data is scanned into a computer and digitized with the use of a software Image J. Since the Image J data is in Pixels, it needs to be scaled in Microsoft Excel to match the original data within ± 1 kHz and ± 1 dB. Particular problems with the plots not having the curves start at the boundaries were overcome with corrections by adding to the scanned curves to correct. As well, the span focus was initially set and adjusted to observe the frequency shift in the highest fidelity that the NA allowed as denoted in the results by Cal6. However, it became important to observe the frequency shift in a directly comparable span as denoted by nocal1A.

In average, the frequency shift caused by removing the cover between spans was + 30 kHz although attenuation was different at 8.3 dB for cal6 whereas 4.1 db for nocal1A as shown in Figure 62 & 63. Differences in the amplitude response or shape between SAW62 and SAW64 were not explainable from the data although believed to be a result of manufacturing variations in the SAWs. Only lab book data was kept for covered SAW64 cal6 data which was used in the calculations above. The conclusion of the tuning process is that it was required due to the frequency shift being $\frac{1}{2}$ of the magnitude of the overall test accuracy. Although, it needs to be mentioned that many tests had an accuracy around ± 7 kHz.

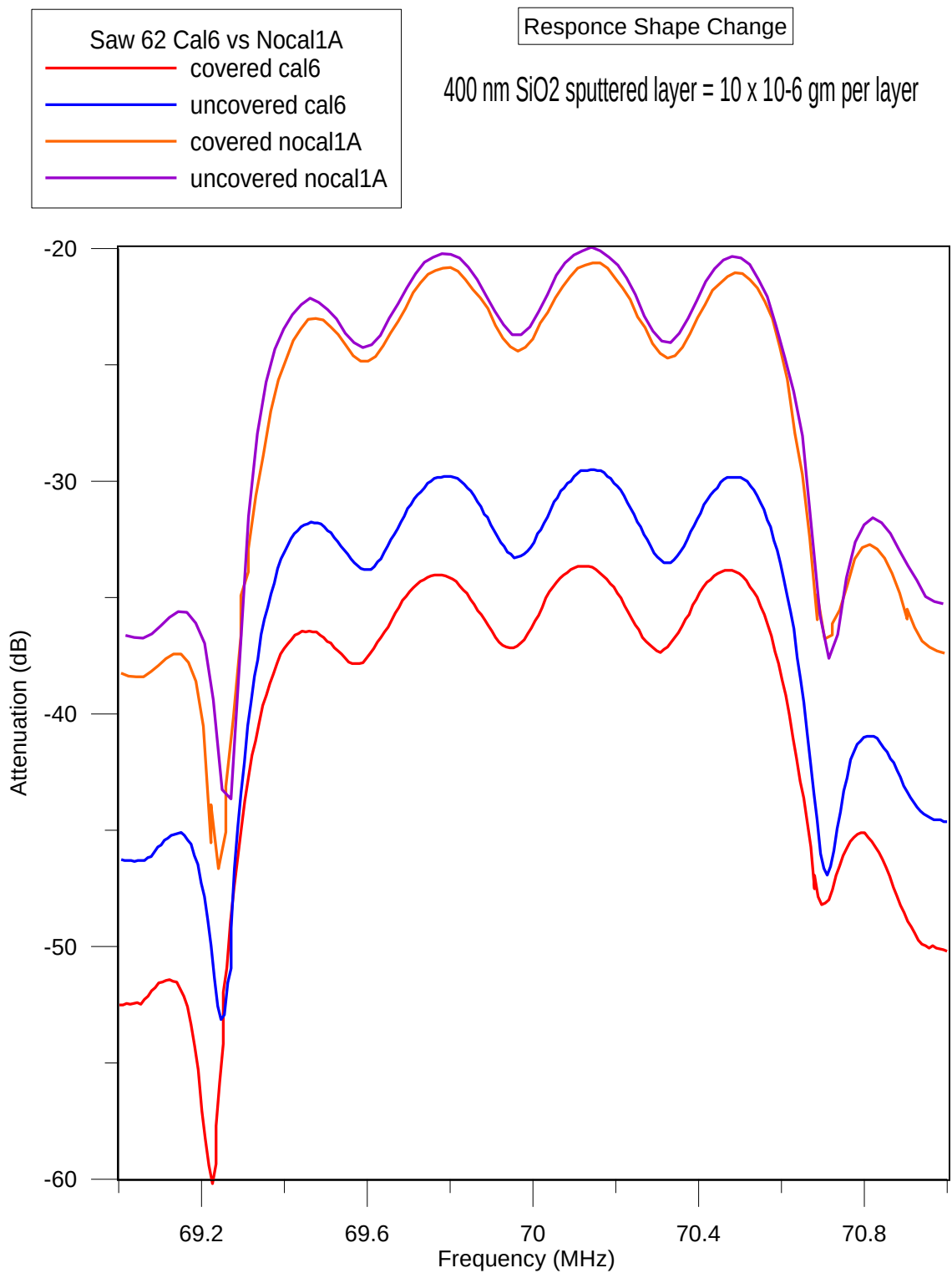


Figure 62. SAW62 Span Repeatability.

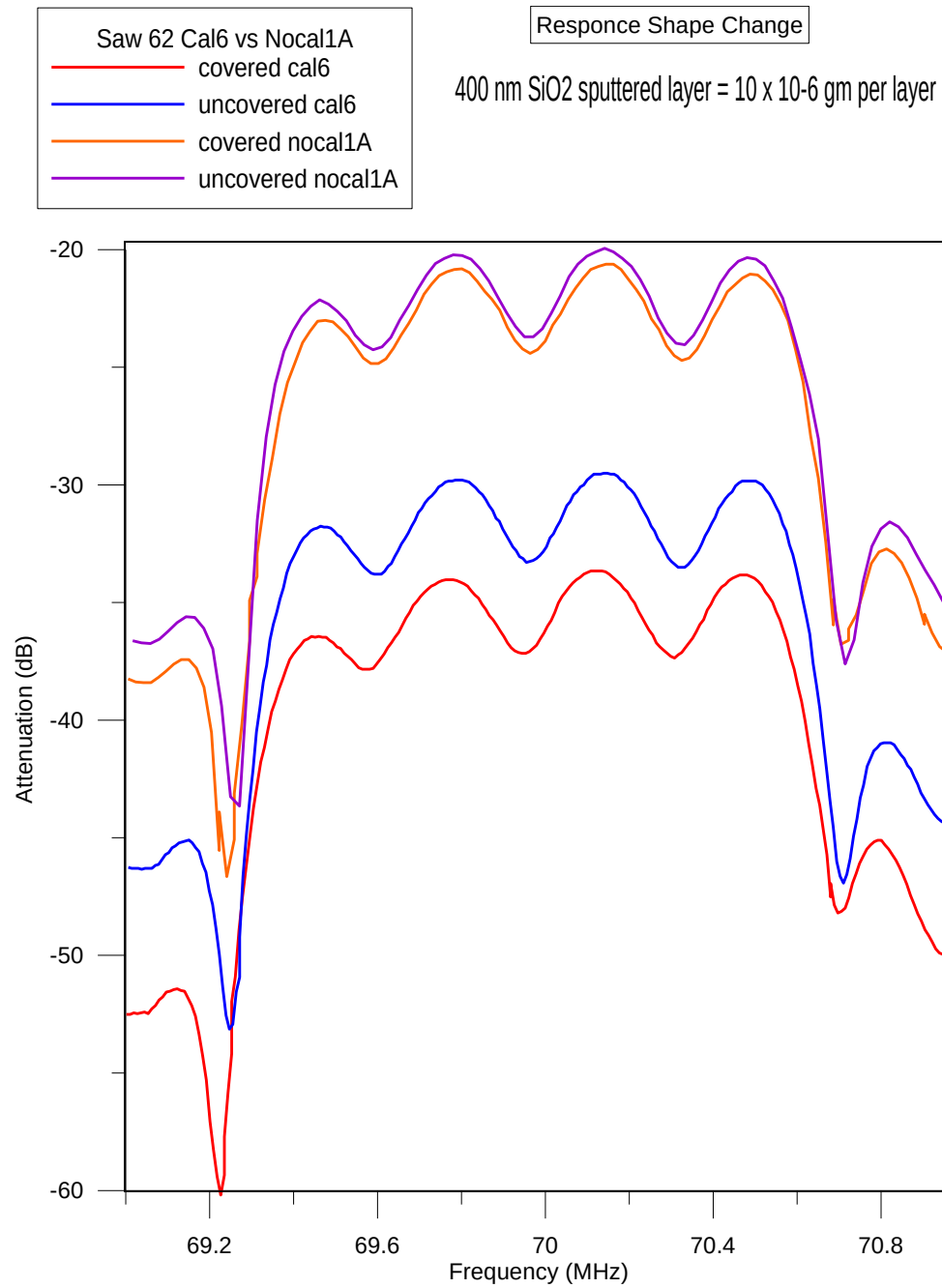


Figure 63. SAW64 Span Repeatability.

Focus was to understand the difference between SAWs based on different span information. A Cal6 span for SAW62 and 64 are shown in Figure 64 and 65.

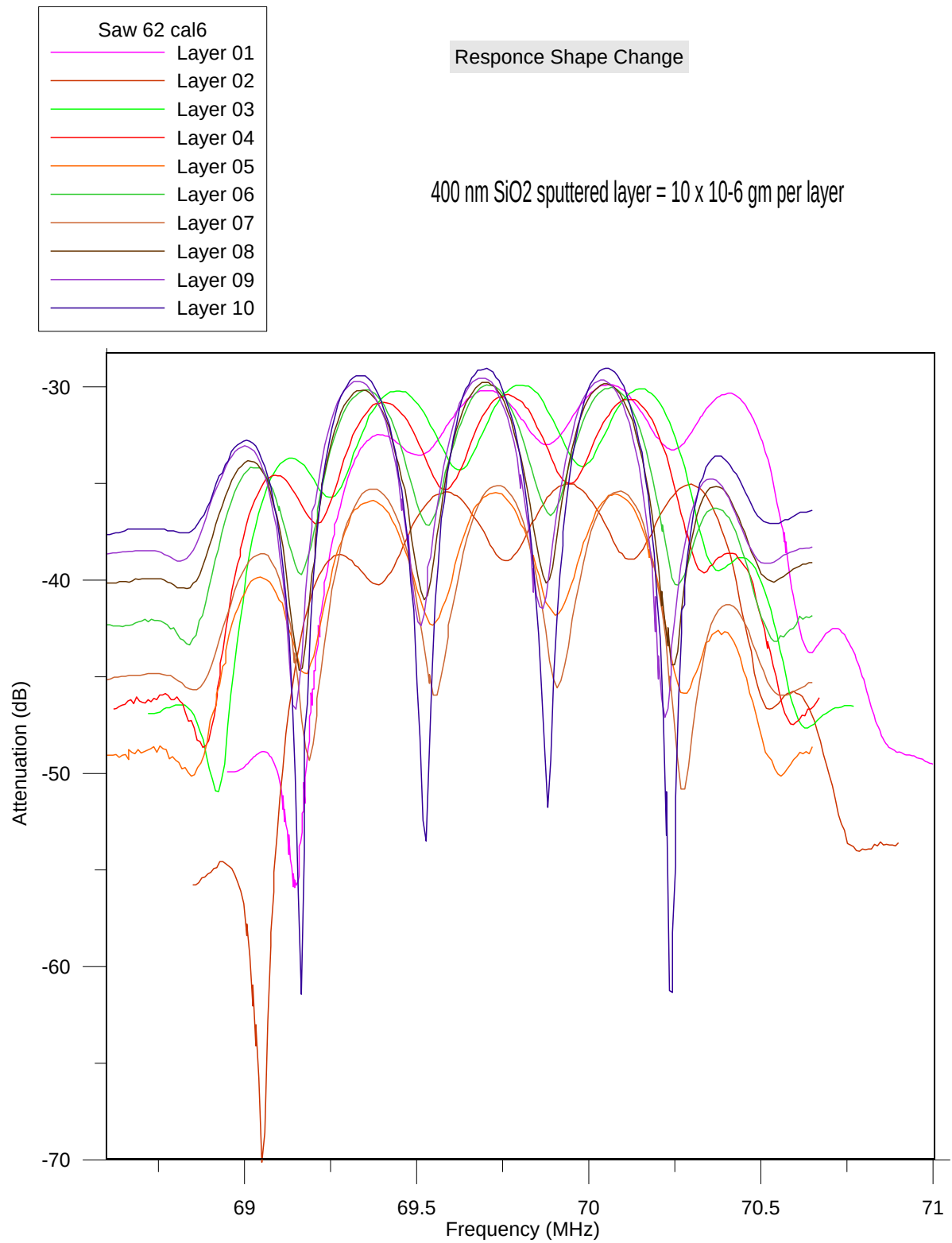


Figure 64. SAW62 Cal6 Comparison.

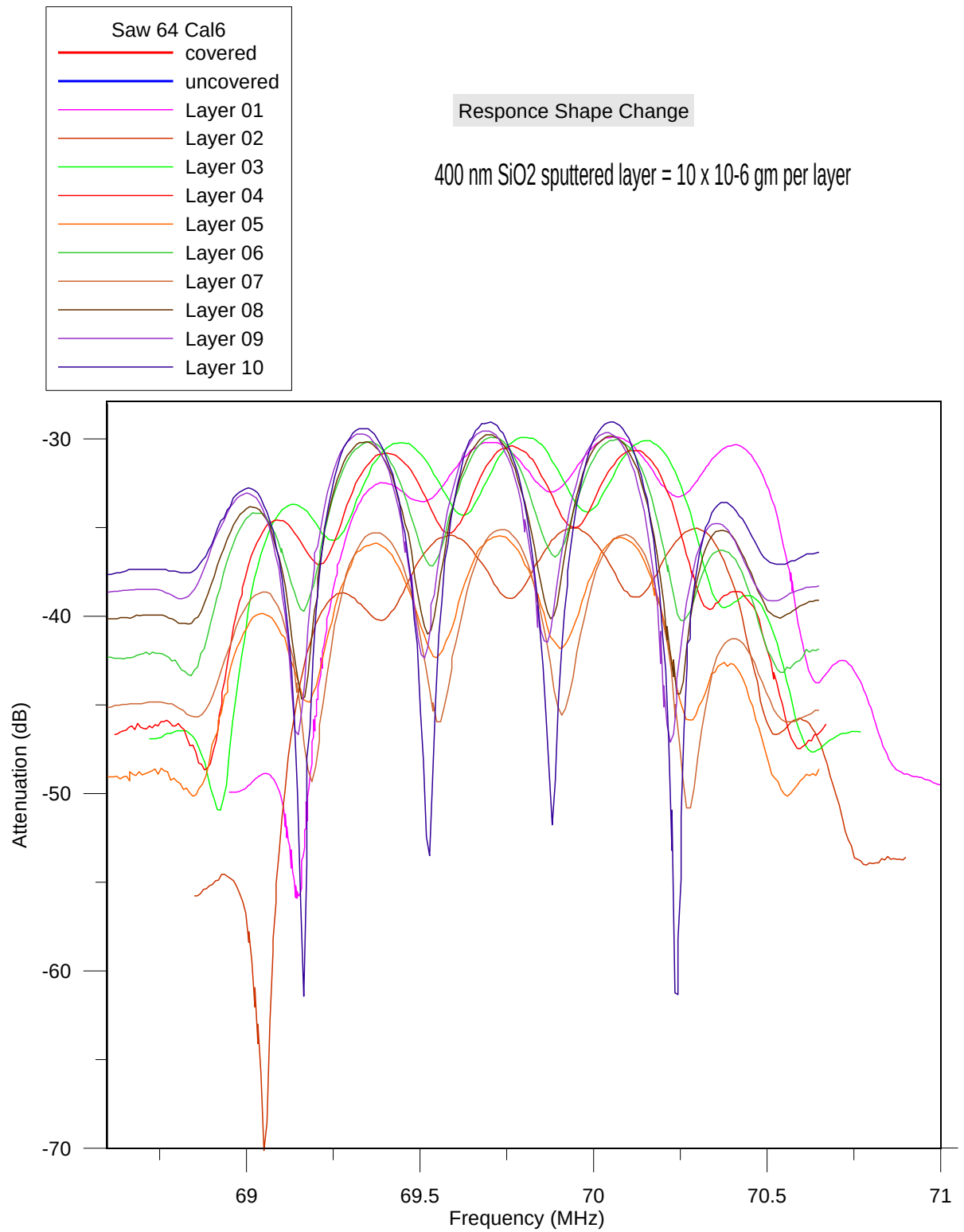


Figure 65. SAW64 Cal6 Comparison.

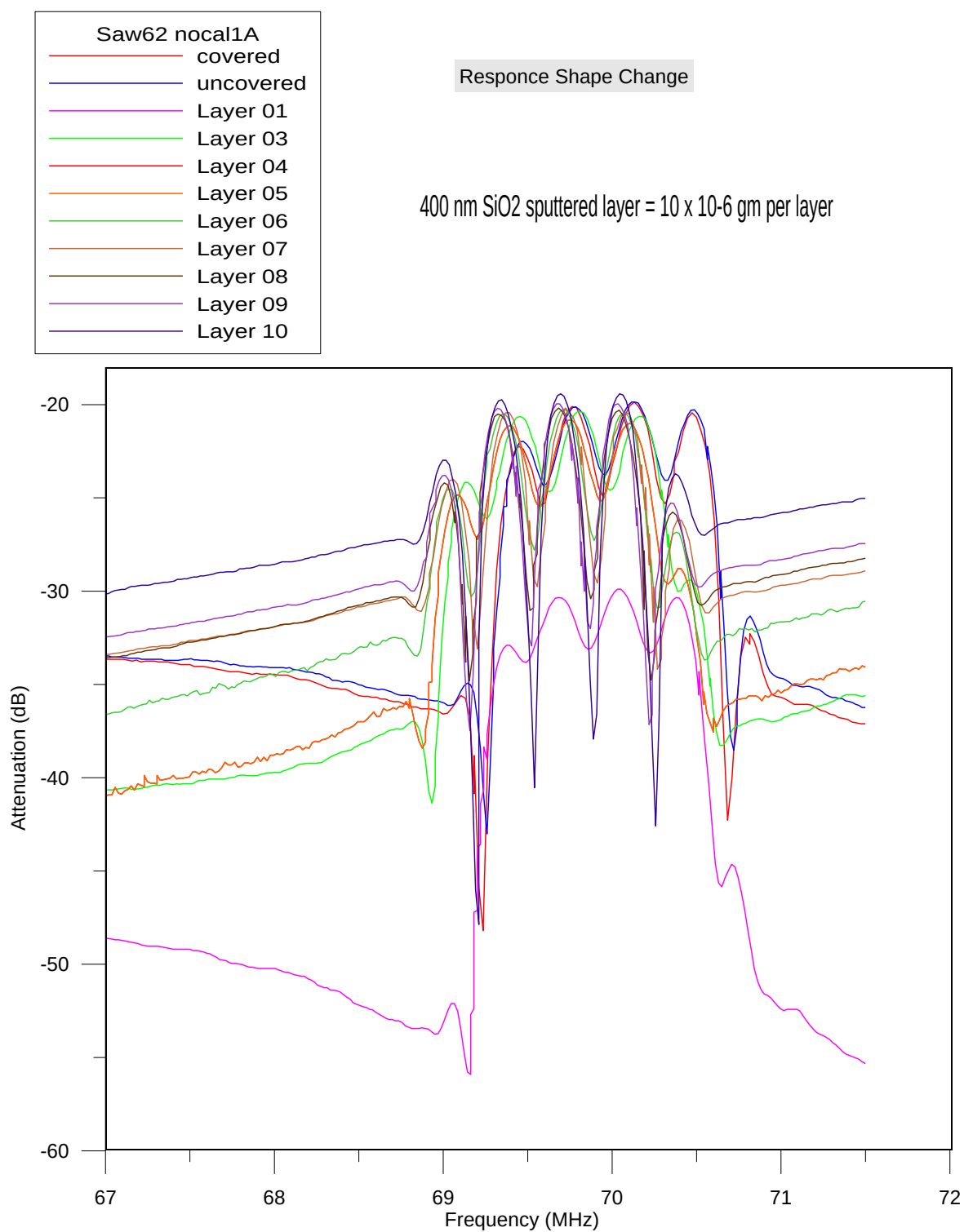


Figure 66. SAW62 nocal1A Comparison.

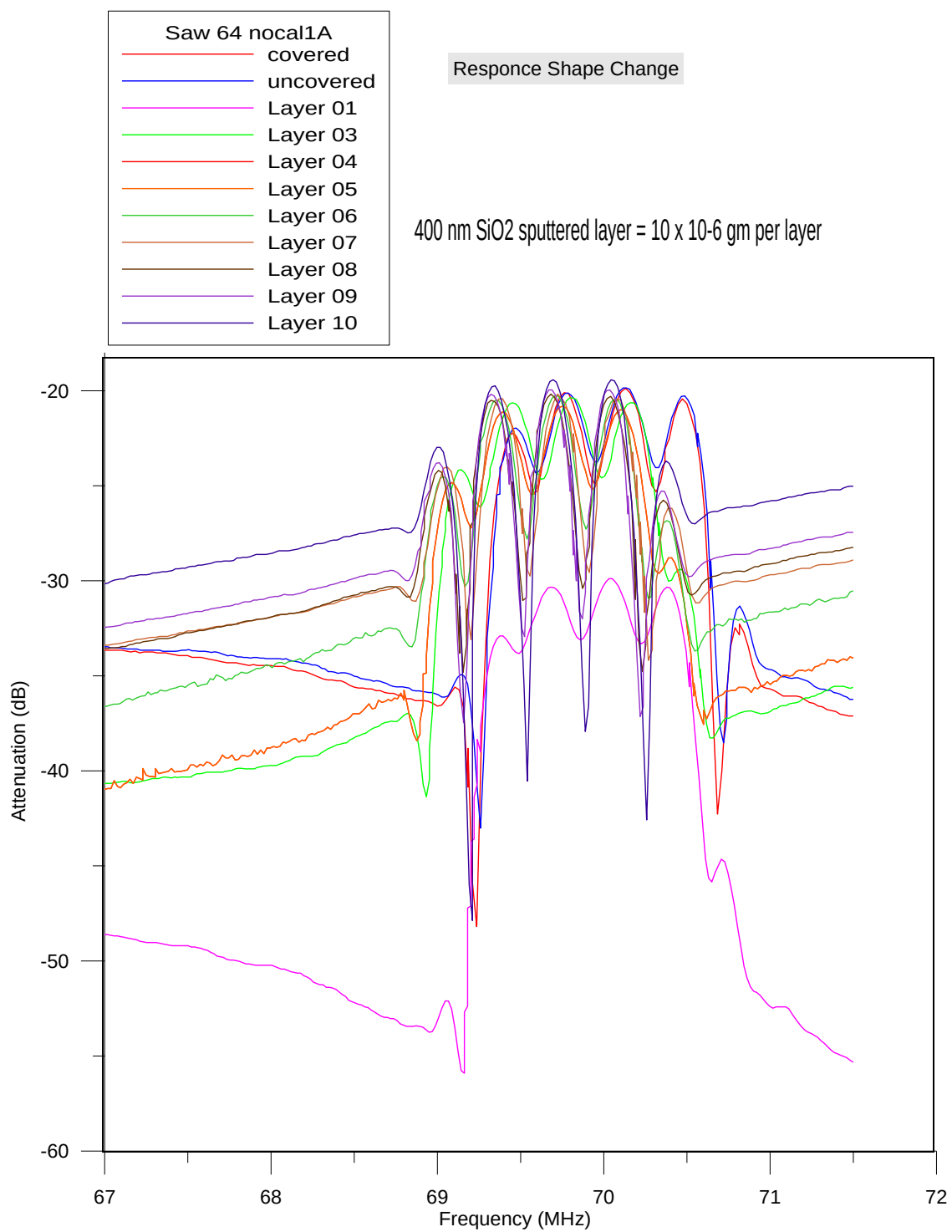


Figure 67. SAW64 nocal1A Comparison.

Interestingly, the comparison between SAW62 and 64 with a span of Cal6 is similar to a span of nocal1A in Figure 66 and 67 which shows the importance of the tuning algorithm to producing results that are repeatable to maximize understanding. The change in slope observed even with direction tracking is due to the output and input of the SAW not being balanced by manufacturer design.

The comparison between SAW62 and 64 will be continued as the data is separated into three distinct patterns. The first pattern in Figure 68 and 69 are with only layer 8 through 10 plotted. The second pattern in Figure 70 and 71 are with only layer 6 and 7 plotted. The third pattern in Figure 72 and 73 are with only layer 2 and 5 plotted.

The first pattern actually reverses the overall trend of more mass resulting in a lower frequency shift. As well, it is easily seen that the damping of signal is also reversed as more mass is added to the SAWs.

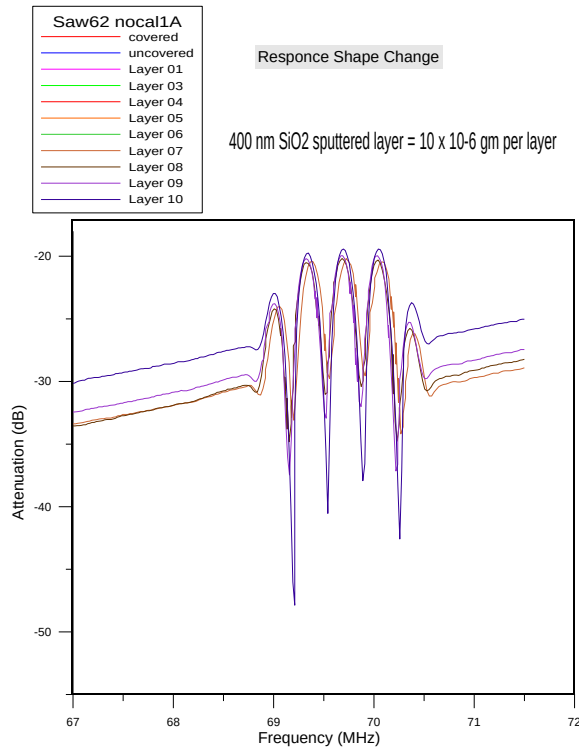


Figure 68. SAW68 Layer 8 to 10.

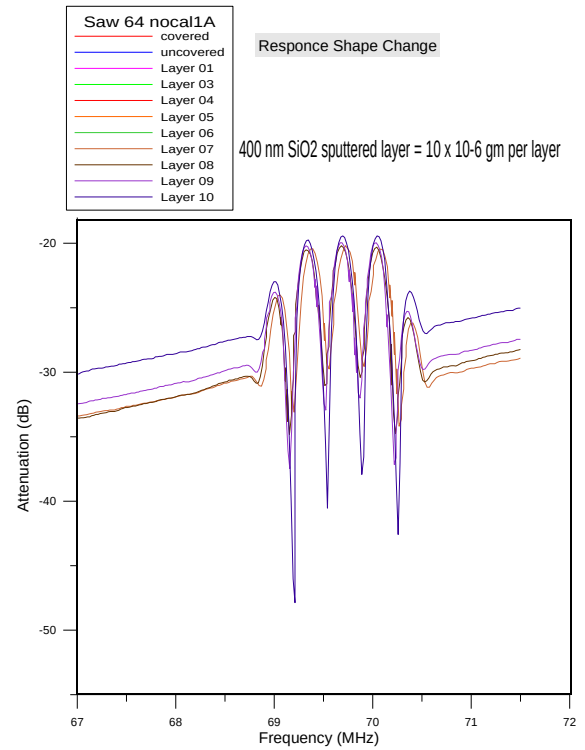


Figure 69. SAW64 Layer 8 to 10.

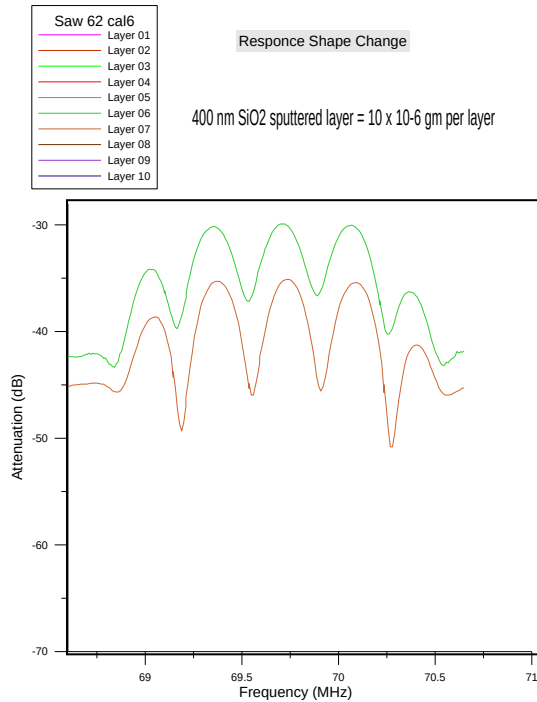


Figure 70. SAW62 Layer 6 to 7.

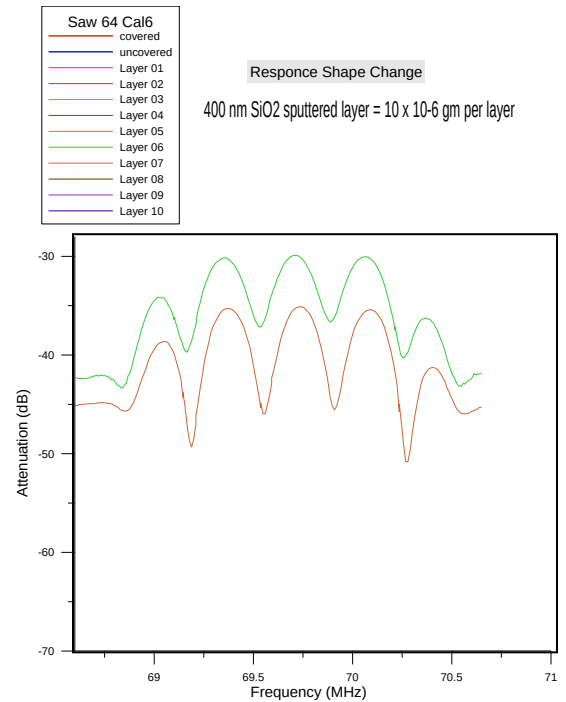


Figure 71. SAW64 Layer 6 to 7.

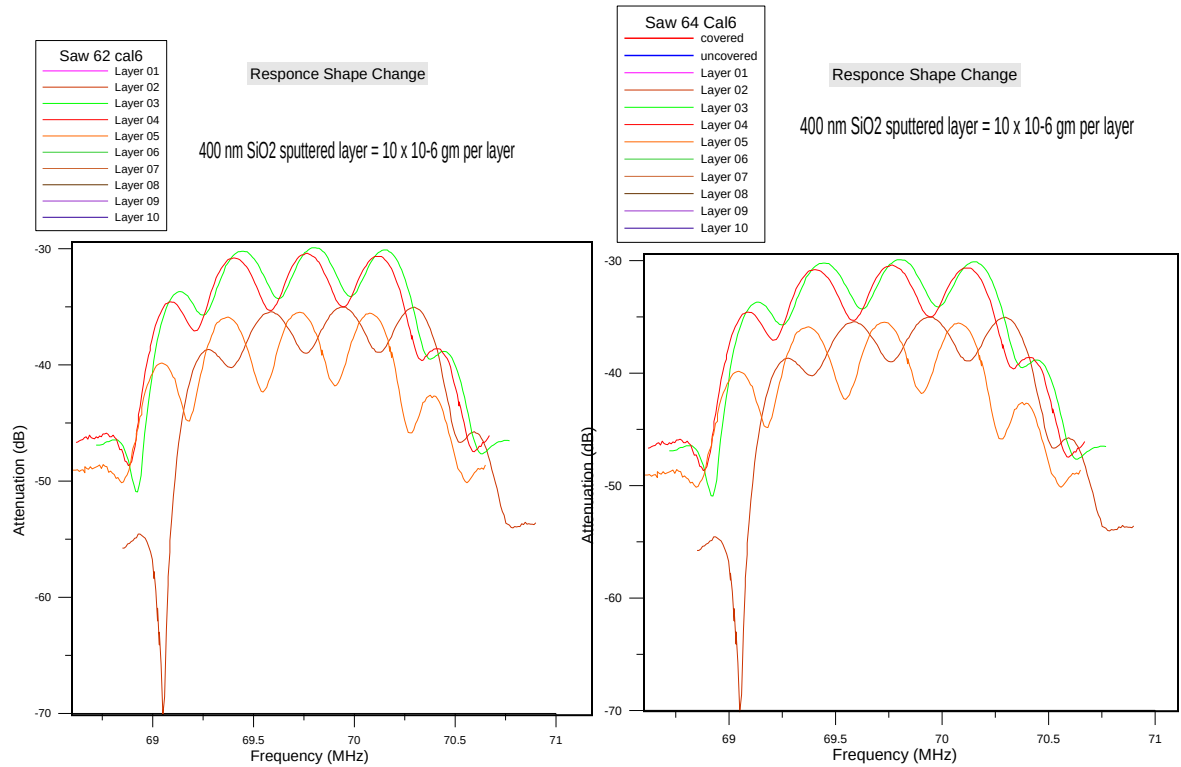


Figure 72. SAW62 Layer 2 to 5 Comparison. **Figure 73.** SAW64 Layer 2 to 5 Comparison

Further understanding was pursued by differencing the responses to understand the nodal response changes. To simplify differencing, an interpolation with Matlab was performed utilizing interp1q command. The comparison of interpolation results to original curves is shown in Figure 74 and 75 below. The resulting difference curves from each surface effect change to the next are shown below in Figure 76. A pattern can be observed in Figure 77 and 78. The data is explained with inter electrode capacitor change as explained in Reference [43] which needs to be correlated too.

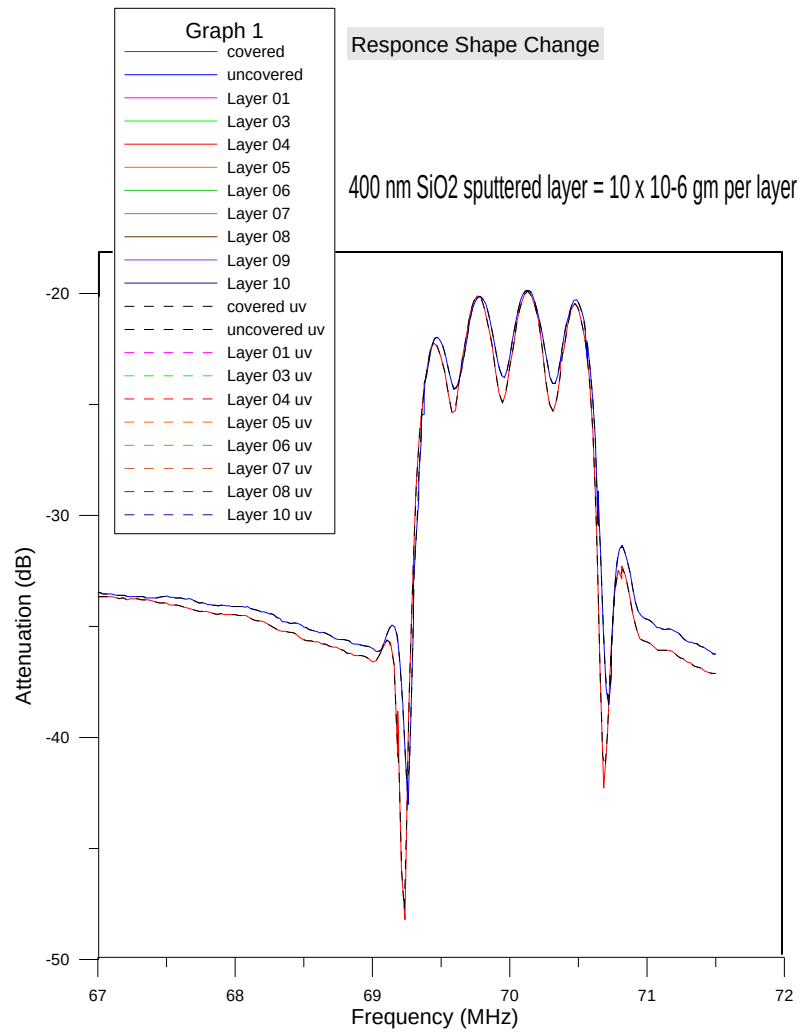


Figure 74. SAW64 Interpolation Comparison.

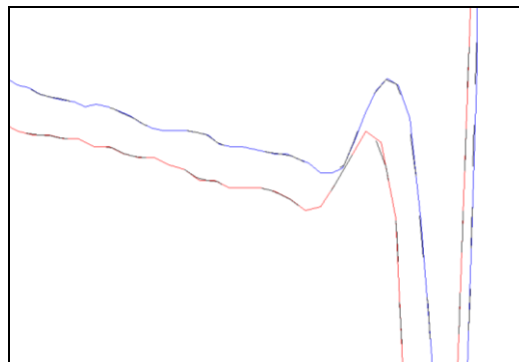


Figure 75. SAW64 Interpolation Comparison Zoomed.

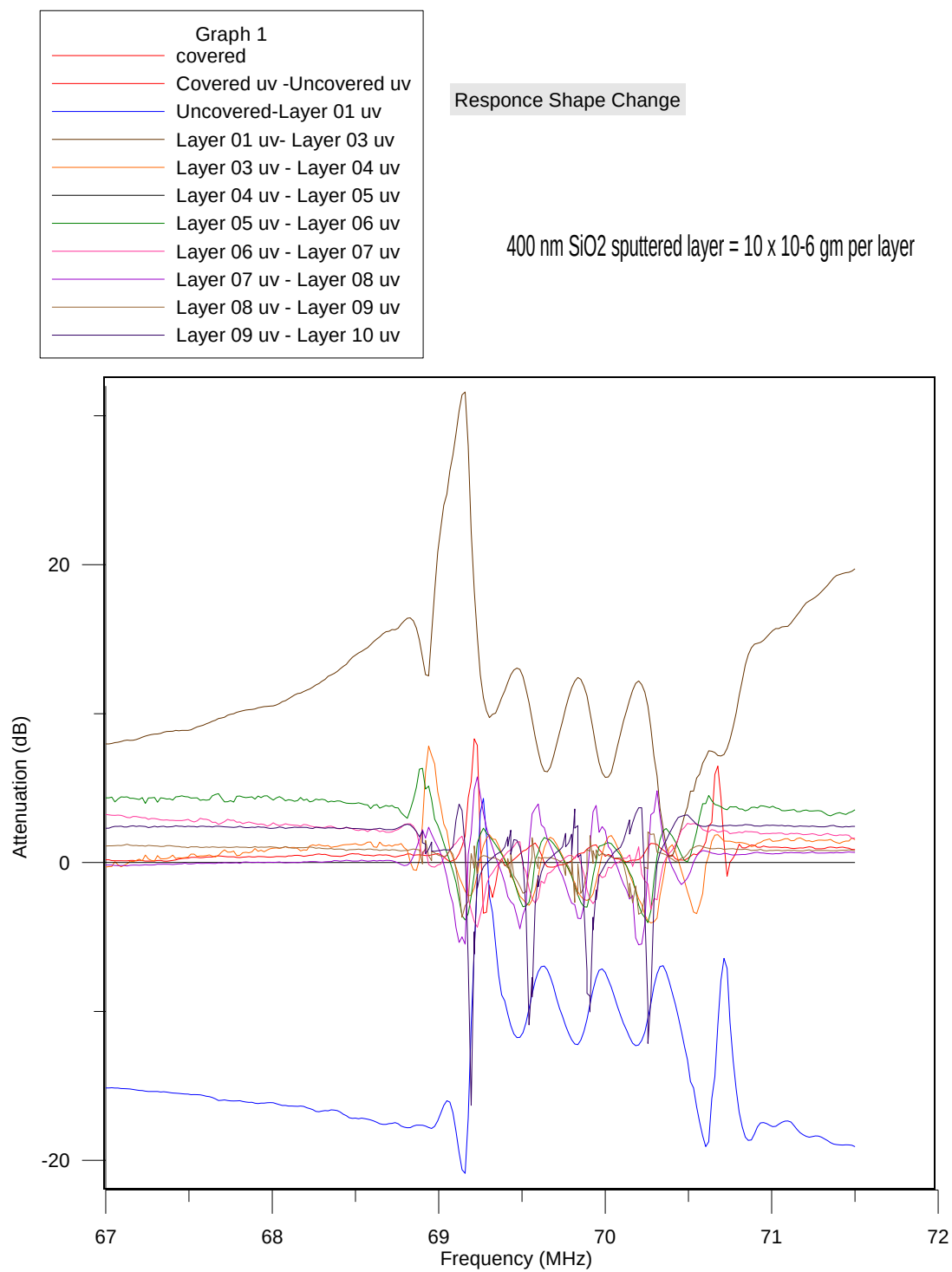


Figure 76. SAW64 Difference Comparison.

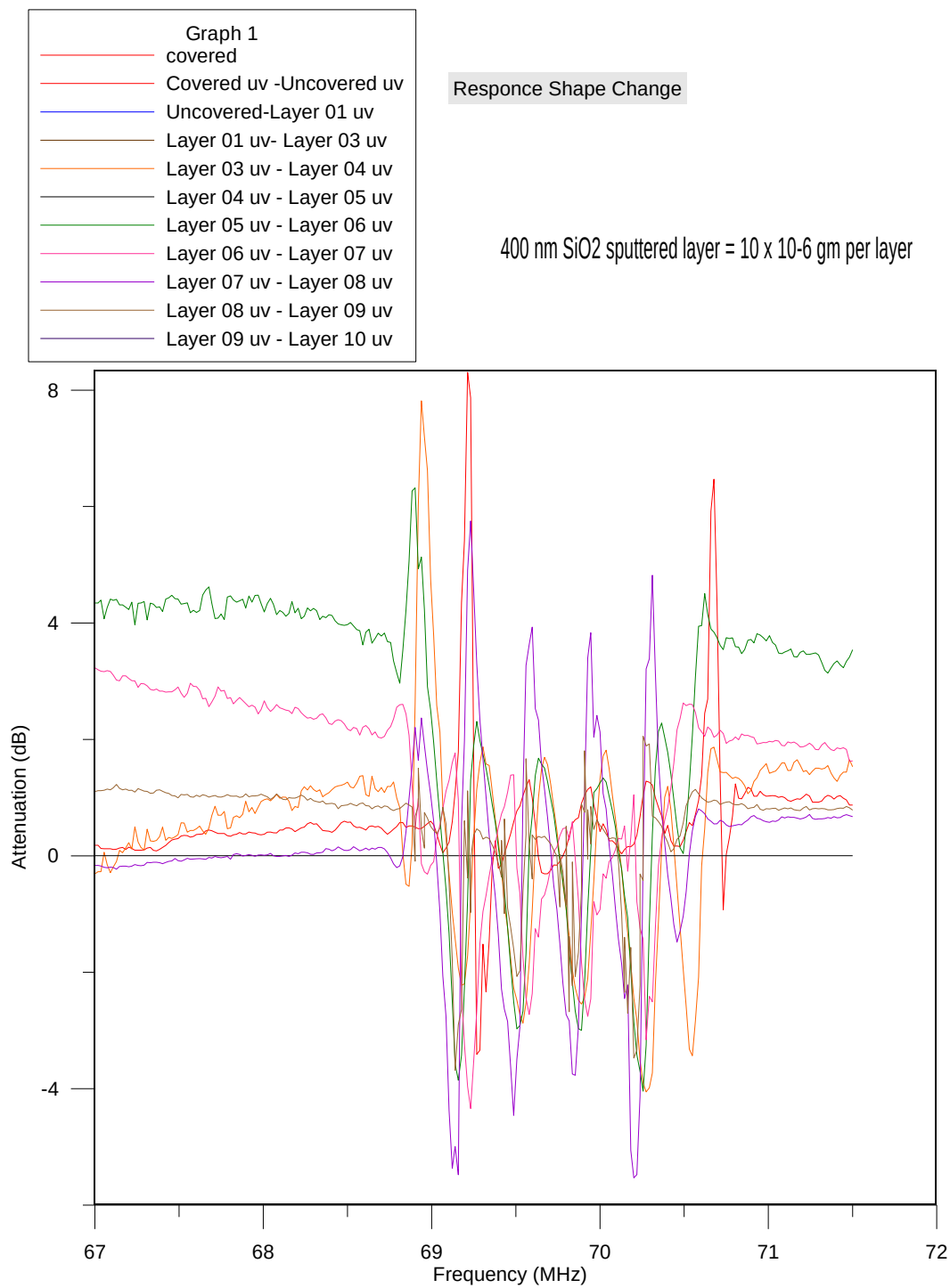
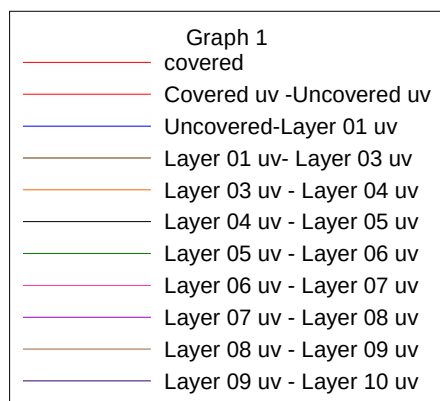


Figure 77. SAW64 Difference Comparison (Pattern1).



Responce Shape Change

400 nm SiO₂ sputtered layer = 10 x 10⁻⁶ gm per layer

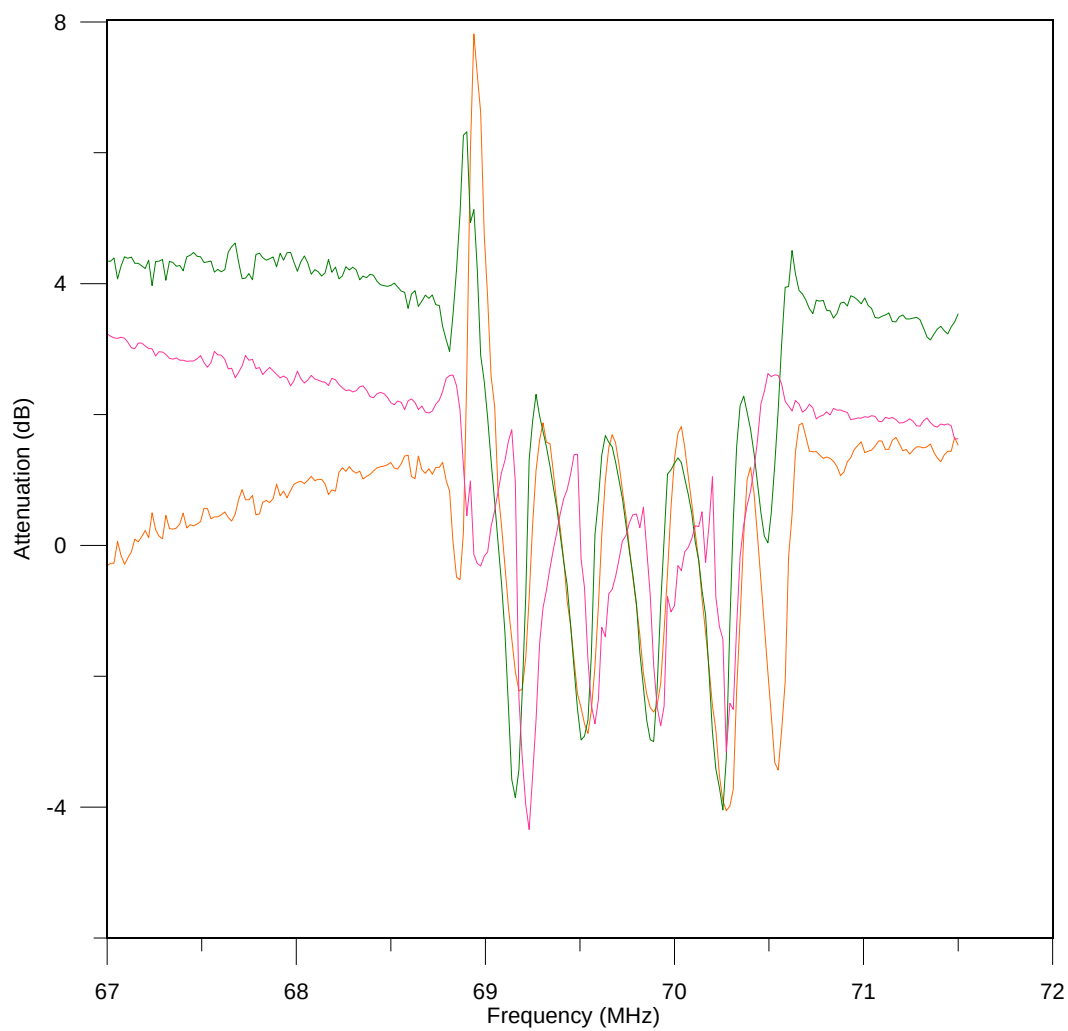


Figure 78. SAW64 Difference Comparison (Pattern2).

4.2 Frequency Shift Analysis of Phase IV Data

These results raise the question of quantification of amount of mass through pattern matching. This researcher thinks this is a premature conclusion with a limited set of films applied. A list of research results is shown below.

1. RFM response appears to match a surface transverse wave device by physical layout. However, manufacturer identifies BP1042 as SAW device. Perturbation models were not analytically correlated to results.
2. Total thickness added by magnetron sputtering is comparable to spin coating technique. The results show that the frequency shift data from the spin coating test was invalid. Hence, no comparison between water glass and SiO_2 was accomplished in this study.
3. More mass equals more damping except in first pattern. Transient response needs to be evaluated to understand some of these findings.
4. Frequency sensitivity gradient appears to support trigger algorithm detection. This indicates that the SAWs studied were significantly sensitive to the mass change applied. A study of the sensitivity response of SAWs needs to be done.
5. Nodal response changes occurred that influence frequency shift detection. A new frequency shift model needs to be developed to correlate these results for an automated detection capability.

6. The quite good replicate data appears to validate the usefulness of trigger based detection with a correlated perturbation model using a SAW with a more complicated response.
7. Static characterization results appear to validate that with a pattern recognition algorithm; the amount of mass and type of mass could be detected with a CDMA SAW.

5. Summary

We have presented a static experimental SAW testing technique that shows the promise of CDMA SAW sensors to quantify mass. We presented experimental results of mass sensitivity for a Code Division Multiple Access (CDMA) Surface Acoustic Wave (SAW) sensor. These experimental results have focused on the static sensitivity of a CDMA SAW sensors assuming that the film thickness is compared to spin coating. A review of research was performed that is focused on dynamic experimental measurement and analytical techniques that are application specific. We have studied a SAW sensor with relatively thick isotropic film layers sputtered on as a static measurement comparison. The results can be used in a detection algorithm to quantify mass on the CDMA SAW sensor. Our results show a set of nodal response wave form changes and frequency shifts due to a continuous mass distribution. The development of the static experimental technique was completed with duplicate SAW tests. The duplicate SAWs had 12 measurements with 10 independently sputtered layers of 100 μm SiO_2 ,

hermitically sealed, and hermitical seal removed using a HP8510A network analyzer. In addition, our results show worst case total error of 0.1% between replicates.

6. References

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The difficulty with website sources is that they disappear and reappear at different locations. This is already the case for several of the discussion sites.

- w1. <http://www3.sympatico.ca/colin.kydd.campbell/> - discussion
- w2. <http://www.sandia.gov/media/acoustic.htm> - technology
- w3. <http://www.sandia.gov/mems/sensors/techinfo/sawgaas.html> - technology
- w4. <http://www.sandia.gov/mems/sensors/techinfo/sawarray.html> - technology
- w5. http://www.thales-tmx.com/html/saw/theory_basics.htm - producer
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