Schnüffeln mit ‘Elektronischen Nasen’:

Supramolekulare Schichten als Diskriminatoren für Chemosensoren.

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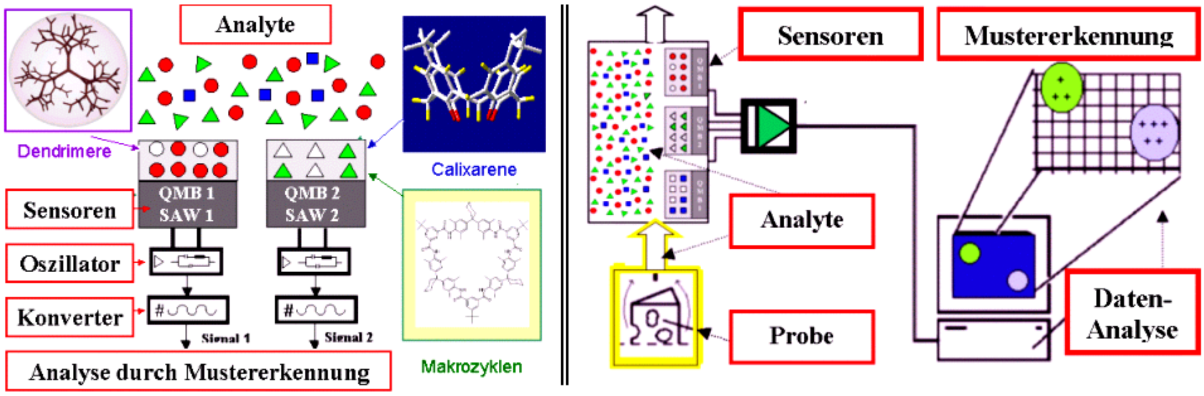
Gravimetrische Chemosensoren, d. h. ’arrays’ oder ’cluster’ aus Schwingquarwaagen (quartz microbalances = QMBs) oder aus Oberflächenschwingern (surface acoustic wave devices = SAWs) auf piezoelektrischen Substraten, sollen chemische Verbindungen qualitativ unterscheiden und quantitativ bestimmen. Hierzu sind diskriminierende Beschichtungen der Einzelsensoren Voraussetzung, die möglichst selektiv und sehr empfindlich mit chemisch sehr ähnliche Molekülen reversibel wechselwirken. Ein chrakteristisches Beispiel für solche Wechselwirkungen sind Einschlußverbindungen auf der Basis des ”Wirt-Gast-Prinzipes”. Hierbei paßt ein ”Gast”-Molekül, - auch als ”Schlüssel” aufzufassen, - mehr oder weniger spezifisch in einen hierzu passenden ”Wirt” wie in ein Schloß, zu dem der Schlüssel paßt.

Als geeignete Wirtverbindungen qualifizieren im besonderen supramolekulare Systeme wie z.B. Dendrimere, Makrozyklen, Calixarene, etc., die im Gegensatz z.B. zu Polymeren monodispers, d.h. gerade auch bezüglich ihrer Molmasse molekulareinheitlich sind, wodurch eine sehr spezifische Wechselwirkung mit Gastmolekülen, - den ”Analyten”, - gewährleistet ist.



Gravimetrisches Sensorprinzip, supramolekulare Diskriminatoren und Datenaufbereitung

Die Erkennung einzelner Komponenten z.B. von komplexen Aromabouquets von Lebensmitteln wie Röstkaffee, Früchte, Käse, usw. gelingt unter Zuhilfenahme von Muster-Erkennungs-Prinzipien, d.h. durch ”pattern recognition”. Als ”übliche” Analyte qualifizieren primär Gase oder leicht-flüchtige organische Verbindungen, (volatile organic compounds = VOCs), aber auch in Flüssigkeiten finden analog konzipierte, aber entsprechend modifizierte Chemosensoren ein attraktives Anwendungsgebiet. Zahlreiche charakteristische Anwendungen für Analyte in der Gasphase gibt es in der Landwirtschaft, z.B. für die Überwachung von biogen emmit-tierten Gasen wie Ammoniak, N_2O, H_2S, etc., aber auch für Konzentrationsbestimmung von Pflanzenschutzmitteln oder Wachstumsinhibitoren bis hin zu Aromamessungen bei reifenden Früchten nach der Ernte. Ferner gelingt die Überwachung und Steuerung von Fermentierungen oder der Silageerzeugung, sowie von Sterilisationen von Pflanzerde oder gar von Plastikartikeln für medizinische Anwendungen (künstliche Nieren, Schläuche für Herz-Lungenmaschinen, etc.). Solche Anwendungenvarianten setzen hinreichend verschieden ansprechende Beschichtungen voraus, wofür supramolekulare Syteme besonders qualifizieren.

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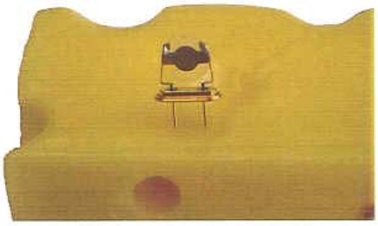

Abb. 2 ***Emmentaler Käse mit Quarzsensor.***

SENSORIK

Von der Waage zur elektronischen Nase

Quarzkristalle können durch Wechselspannung zum Schwingen angeregt werden. Ihre stabile Resonanz macht man sich in Uhren und Mobiltelefonen zu Nutze. Mit wenig Aufwand wird daraus ein brauchbarer Chemosensor.

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Grundlage der Anwendung der Quarzmikrowaage QMB (engl.= *quartz micro balance*) als Sensor ist der Piezo-Effekt. Legt man eine Wechselspannung mit definierter Frequenz an einen Quarzkristall an, so wird dieser im Resonanzfall zu einer Schwingung angeregt (Abbildung 1). Beschichtet man einen solchen Quarz mit einer chemischen Substanz, so erniedrigt sich die Resonanzfrequenz proportional zur Masse der aufgebrachten Schicht. Dieser Zusammenhang ist als Sauerbrey-Beziehung bekannt [1].

Setzt man einen derart präparierten Quarz einem gasförmigen Analyten aus, kann dieser in die aufgebrachte Substanz eingelagert werden (Abbildung 2). Die resultierende Massenerhöhung bewirkt ebenfalls eine Erniedrigung der Resonanzfrequenz, diesmal abhängig von der Konzentration des eingelagerten Analyten. Mit der sensoraktiven Schicht fungiert der Quarz somit als Waage. Um von der Waage zum chemischen Sensor zu gelangen, müssen die Wechselwirkungen zwischen Schicht (Wirt) und Analyt (Gast) selektiv sein. Die Schichtmatrix muss also Hohlräume (Kavitäten) aufweisen, die so beschaffen sind, dass Gasmoleküle des zu detektierenden Gases sterisch möglichst exakt hineinpassen. Außerdem wird der Gast durch elektronische Interaktionen (z.B. Wasserstoffbrückenbindungen oder π-π-Wechselwirkungen) an den Wirt gebunden. In Anlehnung an enzymatische Prozesse spricht man vom Schlüssel-Schloss-Prinzip.

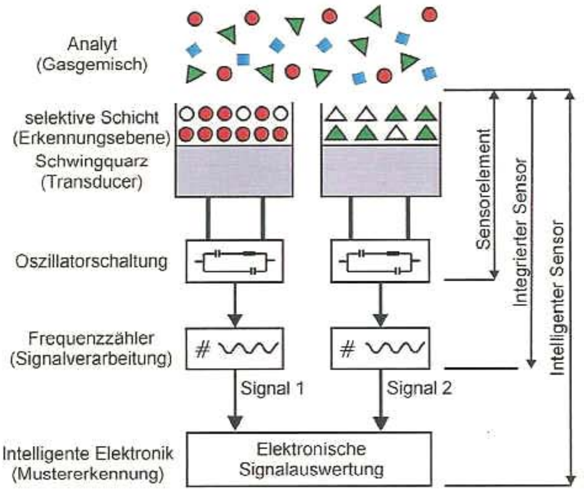

Abb. 1 ***Das Sensorprinzip.***

Damit der Sensor mehrfach verwendbar ist, muss die Interaktion zudem reversibel verlaufen. Die eingelagerten Gasmoleküle müssen sich also wieder herauslösen, sobald die Konzentration in der umgebenden Atmosphäre gesenkt wird. Durch einen „Spül"-Vorgang mit Neutralgas erreicht der Quarz seine ursprüngliche Resonanzfrequenz.

Vom chemischen Sensor zur elektronischen Nase gelangt man, wenn verschieden beschichtete Quarze zu einem Array kombiniert werden. Die einzelnen Sensoren reagieren individuell auf verschiedene Analyte, woraus sich ein komplexes, aber eindeutiges Muster ergibt. Die Analyse des Musters übernimmt ein Computer, wodurch man in der Lage ist, auch vielschichtige Substanzgemische zu analysieren. Die Vorteile QMB-basierter Sensoren gegenüber etablierten gaschromatographischen Methoden sind der geringe technische Aufwand, der hieraus resultierende niedrige Preis, ihre Robustheit und die vergleichsweise kleinen Maße des kompletten Arrays (Größe einer Zigarettenschachtel).

Aktuelles und Anwendungen

Den elektronischen Nasen öffnen sich immer neue Anwendungsgebiete. Eine der ersten praxisbezogenen Bewährungsproben war die Konzentrationsbestimmung von Ammoniak in landwirtschaftlichen Stallungen [2]. Aber auch komplexe Aromen, beispielsweise von Lebensmitteln, können mit einem QMB-Array erfasst werden. Gerade hier zeigt sich eine Stärke dieser Analysenmethode: die Substanzen für sensoraktive Schichten werden in enger Zusammenarbeit mit Spezialisten der supramolekularen Chemie für die jeweilige Anwendung entwickelt und optimiert. Ein gutes Beispiel sind Makrozyklen, die für die Detektion von 2-Heptanon als Leitsubstanz zur Bestimmung des Reifegrades von Emmentaler Käse eingesetzt werden [3] (Abbildung 3). Möchte man eine intensive und reproduzierbare Sensorantwort erhalten, so ist nicht nur die chemische Struktur der auf den Quarz aufgetragenen Substanz, sondern auch das Beschichtungsverfahren von großer Bedeutung. Seine Entwicklung und Verbesserung sind ebenfalls Gegenstand aktueller Forschung und bedürfen interdisziplinärer Zusammenarbeit zwischen organischer, anorganischer und physikalischer Chemie. Kooperationspartner finden sich nicht nur im universitären Umfeld, sondern auch in der Industrie.
Als Beispiel sei die Überwachung der Konzentration von Methylisothiocyanat als Abbauprodukt des Herbizids Basamid genannt, welches von der BASF AG vertrieben wird. Die BASF ist daher an der Entwicklung eines solchen Sensors besonders interessiert.

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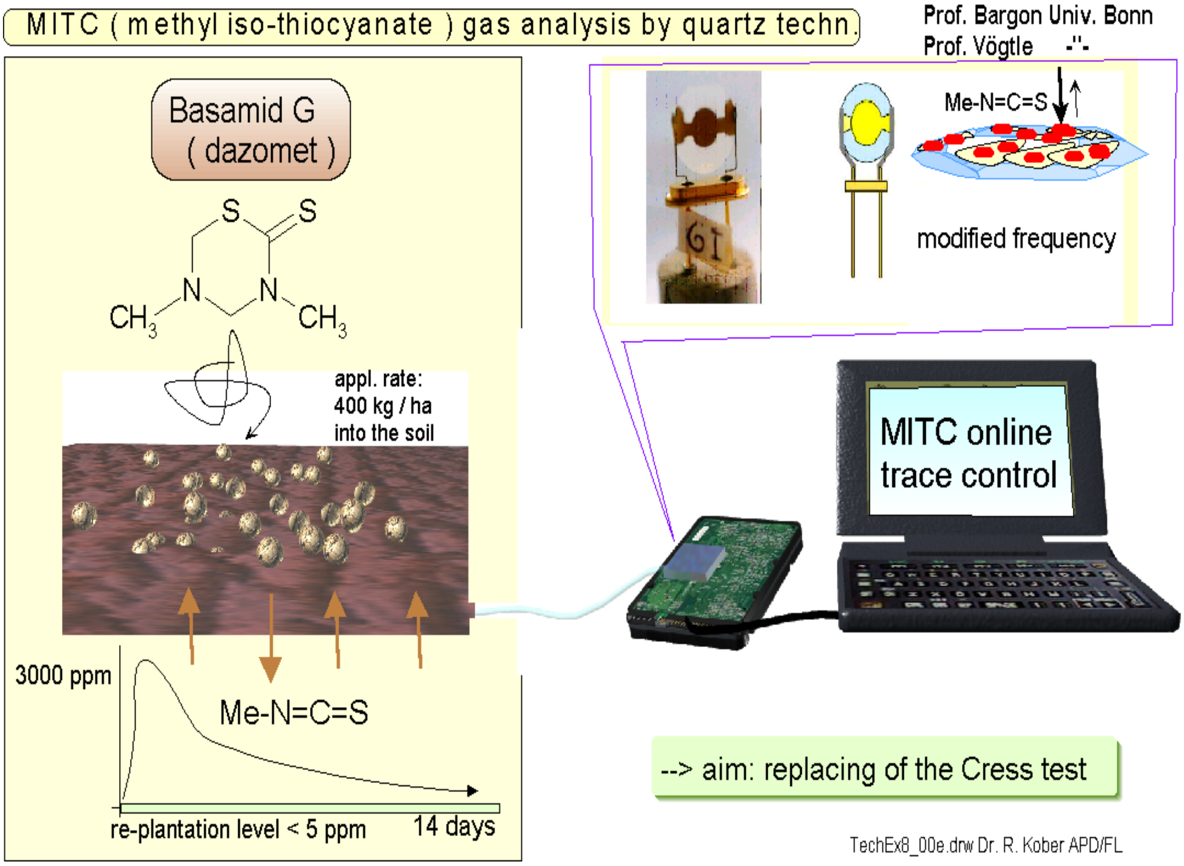
Apendix

Hand-Held Device used by BASF of Germany to monitor the decay of a soil fumigant on line

(essentially similar)



Langasit – a new material class for SAW applications

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Single crystals of compounds with $Ca_3Ga(GaGe_2)Ge_2O_{14}$ (CGG) structure, have recently been intensively discussed as promising new materials for SAW devices. To date the CGG family contains more than 60 members. Since they belong to the same trigonal symmetry group 32 as (low) quartz, similar acoustic properties can be expected. In particular, temperature compensated crystal cuts were predicted. Three additional advantages of these new materials have been discussed so far: (i) The piezoelectric moduli are about three times larger than for quartz. Hence a moderately high electromechanical coupling coefficient is expected. (ii) The SAW phase velocity is lower than for quartz. Hence devices may potentially have smaller sizes. (iii) In contrast to quartz, CGG-type crystals show structural stability up to the melting point. This offers the opportunity of SAW application at elevated temperatures especially in sensing devices. The determination of the density, the piezoelectric, optical, and dielectric material data was reported elsewhere. Here we concentrate on the determination of the elastic constants, of the CGG-type compounds LGS [$La_3\,Ga\,Ga_3\,(GaSi)\,O_{14}$], LGN [$La_3\,(Ga_{0.5}Nb_{0.5})\,Ga_3\,Ga_2\,O_{14}$], and LGT [$La_3\,(Ga_{0.5}Ta_{0.5})\,Ga_3\,Ga_2\,O_{14}$].

We have determined the elastic constants of langasite-type crystals $La_3Ga_2SiO_{14}$, $La_3Ga_{5.5}Nb_{0.5}O_{14}$, and $La_3Ga_{5.5}Ta_{0.5}O_{14}$ from measurements of the sound velocity of bulk and surface guided acoustic modes. The surface acoustic waves and leaky waves were measured by thermoelastic laser excitation .The average measurement error is of about 1 m/s. The angular dependence of the velocity was obtained by mounting the sample on a computer controlled rotation stage. Starting with the elastic tensor determined from bulk acoustic waves we optimised the data set by investigating the influence of the elastic tensor components on the angular dispersion of surface guided waves. This procedure is particularly useful for accurate determination of the non-diagonal elements of the elastic tensor. Acoustic velocity calculations based on the new set of elastic constants show an increased agreement with experimental data compared to the data set derived from bulk waves and previously published material data.

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Acoustic Plate Mode Biosensors

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The identification of specific proteins in a mixture of similar species plays a major role in biomedical diagnostics. Although well-established bioanalytical techniques have proven to be highly sensitive and selective, they usually involve time-consuming multi-stage processes and are, thus, not suitable for on-line monitoring. Moreover, as they are label-based, it has to be guaranteed that the introduction of marker molecules does not significantly change the properties of the biochemical system. In contrast, biosensors facilitate the label-free *in situ* detection of dissolved biological species in real time.

Among the various acoustic wave-based sensor types studied, acoustic plate mode (APM) devices can be designed such that sensor electrodes and analyte are strictly separated. By this means, corrosion processes and short-circuiting at the transducers are eliminated, enhancing the performance and the stability of the devices. In addition, the protection of the electrodes significantly facilitates surface cleaning and preparation procedures. By the use of thinned quartz crystal plates, the lowest detectable immunoglobulin G (IgG) concentration was reduced to values below 100 ng/ml. A major success was the operation of APM immunosensors in complex biological media. In these studies it has been found that the high-frequency devices used are insensitive to cell adsorption, and that antigen/antibody reactions can be successfully detected in both cell/protein mixtures and plasma.

Several design principles have been investigated to improve APM sensor response. Using ZX-$LiNbO_3$ as the waveguiding material, it has been demonstrated that mass sensitivity can be drastically improved by mode coupling between APMs and pseudo surface acoustic waves (PSAWs). However, the high mode densities obtained at elevated frequencies of operation seem to defeat the extension of this concept to the high-frequency regime. In part, the problems of mode interference have been solved by a novel device design, which utilizes different geometries for the wave launching and receiving transducers.

On AT-90°X and –65°Y-90°X quartz, wide-ranging spectra of shear horizontally (SH) polarized APMs have been obtained, which exhibit excellent mode separation and almost identical insertion loss. Using such mode series, surface reactions and material properties can be studied in a relatively broad frequency regime. It has been shown, that frequency effects can be used to distinguish between specifically and non-specifically adsorbed proteins. Furthermore, by appropriately selecting a specific mode from the spectrum, the impact of temperature variations on APM sensor response can be minimized. Such way, the frequency-temperature characteristics (FTC) of the devices have been reduced by almost four orders of magnitude compared to sensors fabricated on ZX-$LiNbO_3$.

Using mode conversion in periodic gratings, a completely new type of acoustic wave sensor has been implemented on YZ-$LiNbO_3$, which combines the advantages of an electrode-free sensing surface – utilized in APM devices – with the high intrinsic mass sensitivity of surface acoustic waves (SAWs). Here, the acoustic wave is directed from the lower to the upper crystal surface, where it interacts with the analyte. Finally, the wave is directed back to the bottom surface and detected at the receiver. The sensor concept has been successfully applied to NO_2 detection in sub-ppm concentrations. A transfer to liquid phase analysis is still being investigated and will require the use of waveguiding materials that support SH-SAWs instead of the Rayleigh type waves excited on YZ-$LiNbO_3$.

Chemical Sensors:
A comparison of QCM and SAW devices

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Mass-sensitive devices are of pronounced importance in chemical sensing, since they are transducers which give a response to every analyte. Well known are QCMs (quartz-crystal-microbalance) and SAW (surface-acoustic-wave) resonators and delay lines. Usually, QCMs are operated at 10 MHz up to 40 MHz and show a mass sensitivity down to one Nanogram, whereas SAWs have resonance frequencies between 100 MHz and 1 GHz leading to a limiting sensitivity of one Picogram.

Due to the high sensitivity of the SAW surface phenomena are very important. A cross sensitivity to humidity is observed at gas phase measurements. Swelling leads to a change in material properties, even a frequency enhancement is possible and volatile organic compounds (VOCs) give a competitive solvation. These surface effects can be utilized via a covalent linking of molecular cavities (calixarines, cyclodextrines) in host-guest chemistry. This molecular recognition allows the detection of halogenated and aromatic hydrocarbons, down to a few ppm, with monolayers and is superior to the QCM according to the signal to noise ratio.

Favourable bulk effects in gas phase detection can be evoked by molecular hollows and molecular imprinting. Selectivities achievable in this way for lean molecules are better than with natural antibodies. These synthetic antibodies are suitable in layer heights from 40 nm to 100 nm as sensitive coating for SAWs in the frequency range from 75 MHz to 1 GHz. According to the prediction of Sauerbrey a parabolic increase in the frequency response is observed. The signal to noise ratio increases approximately in a linear manner. Thus, 0.1 ppm of aromatic and halogenated hydrocarbons are monitored with a S/N of 3:1. This influence of the device operating frequency can also be deduced from SAW and QCM measurements, where the same coating height yields a detection limit of 0.1 ppm for the SAW and 4 ppm for the 10 MHz QCM (e.g. aromatic hydrocarbons).

Mass sensitive devices in the shear mode are also operable in solution, even complex motive oils can be characterized. In spite of the considerable damping in solution QCMs are highly sensitive sensors. Even high frequency SAWs are suitable for liquids and the sensitivity benefits from frequency. Quartz devices can be applied in liquids with a low dielectric constant, while lithium tantalate is applicable for aqueous systems. Some problems arise, however, since the decisive temperature dependence is given not by the viscosity of the medium but by the substrate.

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Resonators and Delay Lines: A Comparative Study of Surface Acoustic Wave Sensors (Resonatoren und Verzögerungsleitungen: SAW-Sensorik im Vergleich)

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Abstract – This contribution aims at surveying surface acoustic wave (SAW) delay lines and resonators for sensing applications. First, the fundamental characteristics of the SAW and means for their technical exploitation are shortly discussed. The concept of the interdigital transducer (IDT) as an electro-acoustic antenna is introduced together with two modeling approaches, viz., the delta-function and the hybrid (P-)matrix method. This enables one to explain the terminal characteristics of the SAW delay line and to understand circuit effects such as the amplitude ripple in the frequency response due to multiple-transit signals.

The SAW one-port resonator, which consists of an IDT placed between two distributed (Bragg-type) reflectors, is thoroughly discussed from an equivalent-circuit point of view. Formulas for the resonator admittance, the resonance and antiresonance frequencies, and the quality factor *Q* are derived from an analysis of the equivalent circuit. A Fabry-Perot model allows one to link the equivalent-circuit parameters to the underlying physics. This is used to deduce some basic design rules and to explain the existence and effects of longitudinal and transverse modes. It is also shown how the concepts may be expanded to obtain a two-port resonator.

Further considerations are devoted to the origin of sensor effects on SAW devices. Some typical effects and material parameters are presented, and possible sensor instrumentation approaches are exemplified by oscillator circuits.

In the final part, the respective strengths and weaknesses of sensor delay lines and resonators are compared with each other. Criteria applied include general design issues, the amplitude and phase responses, chemical and biological applications, oscillator design, and wireless sensing applications. As it turns out, delay lines offer more design freedoms than resonators and can be operated more easily at higher frequencies and in liquid environments. On the other hand, up to about 1 GHz, resonators result in lower-noise oscillators and consequently in a higher resolution. None of the above features warrants a definite decision in favor of either design.

Gez.

Prof. Dr.-Ing. Gerhard Fischerauer

QCR-SENSORS –MODELS AND APPLICATIONS

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Bulk acoustic wave sensors are sensitive, to varying degrees, to perturbation of many different physical or chemical parameters, both intrinsic to the device and extrinsic. Chemical/biochemical sensitivity is typically imparted by attaching a thin film to an acoustically active region, e.g. a region having significant acoustic amplitude, of the acoustic device. Acoustic sensing is only possible when this film or the adjacent medium interacts with the acoustic modes. Commonly, the increased mass density of the film, arising from species accumulation, is relied upon for a sensor response. Such behavior occurs in the so-called mass-sensitive devices. Changes in other parameters, including elastic, electrical, mechanical or other properties, can also considerably contribute to the sensor response. They can be described by the non-gravimetric effects. In last years different models were developed to understand these effects and their influence on the sensor response. The understanding of the transduction mechanisms of acoustic-wave-based sensors is crucial for a pronounced progress in their industrial and scientific applications.

QCRs (quartz crystal resonator)s are considered more in detail as an example for manifold used bulk acoustic wave resonator sensors. Selected application examples will be presented.

QCR- general description

The quartz crystal resonator (QCR) is the most common device used as acoustic-wave based sensor. The simple geometry of the device and the predominant thickness-shear mode of the propagating wave are propitious conditions for a comprehensive derivation of the acoustic-electrical behavior of quartz crystal devices. Acoustic wave generation and propagation is most concise, therefore a coated QCR is used here as example to demonstrate the physical background of acoustic-wave-based sensors. Other acoustic microsensors have a more complicated wave propagation pattern; the concepts of coated piezoelectric resonator modeling are the same. The analytical approach to describe acoustic wave generation and propagation in a QCR sensor is based on the linear piezoelectric equations together with Newton's equations of motion and Maxwell's equations. A chemical or biochemical QCR sensor can be understood as a multilayer structure of one piezoelectric layer and a certain number of non-piezoelectric. Different cases can be distinguished, e.g. a quartz crystal with a single film as the simplest example of a QCR gas sensor, or a quartz crystal coated with a single film, which is in contact to a liquid as a typical biosensor arrangement. The penetration depth of an acoustic shear wave into a liquid is very small, e.g. 250 nm in water at 5 MHz, therefore the acoustic wave decays before reaching the surface of the liquid and almost any liquid film can be treated as semi-infinite. Different models like transmission line model(TLM), BvD-model and acoustic load concept(ALC) are introduced and the consequences for sensor applications are discussed.

Applications

Two different kinds of systems are used as sensor interface electronics for the determination of frequency, phase and damping changes of the QCR sensor. On the one hand, there are expensive, voluminous network analyzers, which are applicable for laboratory analysis and research. An alternative concept of a miniaturized network analyzer is shown. On the other hand, there are oscillator circuits with a quasi-digital output signal . The application properties of both systems are compared. Selected examples of using QCRs for chemical and biochemical sensing are presented. Acoustic devices are now established in chemistry as one analytical method. The possibilities and limitations of using this transduction principle for a (bio)chemical analysis are determined by the sensor itself, the sensitive layer or adjacent medium and the sensor interface electronics.

The classical Sauerbrey-equation provided the theoretical base for most successful industrial application of QCRs up to now in the form of the DuPont humidity sensor system and in different monitoring systems for the observation of deposition processes, e.g. metal deposition. In the last years many attempts were undertaken to put new products on the market which use bulk acoustic wave sensors. Thus QCRs were applied in a few "electronic nose" systems or in commercial QCR system for the investigation of liquids.

A CONTACT-FREE SAW BASED TAGGING SYSTEM FOR HARSH ENVIRONMENTS

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Keywords: *Tagging system, SAW, harsh environment*

Driven by the need for identifying an AVL pressure sensor inside of a combustion engine, a tagging system based on a SAW identification tag (ID-tag) is introduced. The tagging system is compatible with the constraints placed by both the harsh environmental conditions acting on the pressure sensor mounted close to the combustion chamber and the reading technique employed to monitor the data. The system is stable up to at least 400 °C for a long period in time, resistant against high temperature gradients and withstands external shocks or vibrations. The tagging system is attached contact-free and fits into a small size bore so that neither the shape of the pressure sensor nor the cable has to be modified. Up to 10^5 different sensors are addressable on such a small-sized SAW ID-tag providing every pressure sensor with an individual number. These numbers have been successfully read out up to 400°C, and owing to the intrinsic temperature dependence of the reflector positions on the tag, their analysis also allows to account for a measure of the temperature within the pressure sensor. Furthermore, the solution copes with additional demands such as low-costs as .a cheap reader unit has been developed, too. Based on this present work a technique that allows identifying any kind of sensor read out by a hf-compatible cable appears to be feasible.

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Visualization of the near field of airborne sound generated by loudspeakers and parametric arrays using TV holography

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Parametric arrays especially built for applications at high audio sound pressure use the most effective ultrasonic transducer for airborne sound, i.e., a piezoelectric (PZT) bimorph. Since the individual transducer elements are very small (<10 mm in diameter) several hundred of them have to be combined to reach the desired audio sound pressure level. Because each element has a slightly different resonance frequency causing a large electrical phase shift at the operating frequency, the resulting ultrasonic sound field near the array is of special interest. The very small variation in the refractive index of the air caused by the pressure variations of the propagating sound wave can be used to study the sound field by an optical method called TV holography [1]. It was demonstrated how sound fields of loudspeakers, of parametric arrays and of single ultrasonic transducers can be observed and recorded in real time by use of time-averaged TV holography with sinusoidal phase modulation for increasing the sensitivity. The recordings represent a two-dimensional projection of the sound field integrated along the viewing direction. By image processing combined with phase shifting technique the integrated amplitude and phase distribution of the sound field can be calculated. In many cases these two dimensional projections already give valuable information. The capability for the complete description of the three-dimensional sound field is given by recording along different directions and tomographical backprojection [2]. This feature will be incorporated into the system for our future work.

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Modeling of Sensor Function for Matched SAW Resonators

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Abstract:

SAW resonators are particularly suitable for passive wireless sensing because of the high Q factor (storage of rf energy) and the possibility to change their resonance behavior influenced by an external measurand (frequency pulling).
To ensure a good performance, such passive systems have to be carefully designed for energy efficiency and for the demands of the interrogation regime.
For well designed sensors, the parameters of equivalent circuitry (PEC) of the resonator have to be estimated with a high accuracy, in order to simulate the resonator pulling in combination with a matching network.
Hereby, the matching network does not only match the resonator – it transforms the whole resonance behavior and influences it. So a sensor is constructed by the implementation of a sensing element (e.g. variable capacitor) into the matching network.
Based on measured data of different resonator types, the PEC have been precisely calculated by an improved method with Hilbert transform. The PEC model is extended by a matching network to simulate the interaction between the two parts, which leads to a modified resonance behavior. Consequently, different matching circuits have been calculated and measured for the applicability of pulling the resonance frequency inside a reasonable region of mismatching.
For practical purposes, an approximation of the sensing function and the energy loss through mismatching is possible.
This paper discusses the simulations, which have been verified by experiments.

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Advantages and applications of non-linear sub-bottom profilers

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The ideal sub-bottom profiler for many survey tasks should have among others a narrow beam width and no side lobes, a high bandwidth and transmitting pulses without ringing as well as high mobility and therefore small and light equipment. Some of these desires are only to fulfil by using non-linear acoustics.

INNOMAR'S parametric sub-bottom profiler systems SES-96 and SES-2000 provide not only the exact water depth determination, but give also detailed information about sediment layers and sub-bottom structures with very high resolution. The systems use primary frequencies around 100kHz. The half power beam width of the transducer at transmitting is only +/-1,8°. Due to the principle of non linear acoustics this small beam width is also valid for the generated low frequencies between 4kHz and 15kHz. The small beam at low frequencies gives a never seen horizontal resolution and the possibility to detect embedded small scaled objects or structures.

Additionally it is possible to generate really short signals, as for example 1 wave of 15kHz without ringing, and therefore to achieve a high vertical resolution of sediment structures or objects.

By using of multi-frequency signals the estimation of the attenuation coefficient – one of the important parameters to describe sediment properties – becomes possible with reliable results.

The non-linear sub-bottom profilers of INNOMAR are used worldwide for a great variety of shallow water applications from 1 meter down to 1500 meters of water depth. The compact design of these family of echo sounders, only the transducer and a water protected 19”inch unit are required, allows very easy and mobile installations. The accuracy of the depth measurements fulfils the IHO special standard and the high vertical and horizontal resolution on-line results of the low frequency channel are significantly higher than with any linear system on the international market.

Due to the small beam width also the electronic beam stabilization and beam steering becomes possible. This might be useful especially for applications offshore and under rough survey conditions. Additionally the search for embedded objects will be more effective.

Typical applications in the marine field are for example geological/ geophysical investigations, determination of mud thickness and sand search especially for dredging tasks, cable or pipeline route surveys for the offshore industry and searching for pipelines and embedded objects.

Design and Characterization of SAW Biosensors

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Biosensing with acoustic surface waves encounters several technical problems which shall be shortly presented here together with approaches for their solution. They are mainly caused by the fact that biological reactions occur in liquid media like buffer solution or biological ones like blood or sera. All of these liquids are aggressive against the metallisation of the commonly used SAW devices.

The main problems are:

- The excitation of bulk waves in the sample liquid, leading to an overdamping of the surface wave. A remedy can be found by employing shear modes like, e.g., Lamb waves that are guided at the surface by a polymer or glass layer of roughly 1 µm thickness which can also be used to protect the metallisation from corrosion.
- Dielectric or ohmic short of the transducers by the conductive sample liquid. This effect can be avoided by the use of high permittivity substrates (e.g. $LiTaO_3$) and by the low-permittivity guiding layer mentioned above.
- Influence of the sample conductivity onto the SAW velocity. In order to suppress this effect, a metallization is evaporated onto the delay path of the SAW device. This film can also be employed for immobilization of the protein layer required for sensor operation.
- Conventional contacting of the SAW device with bonding wires impairs their handling and causes problems because they must be protected from the sample liquid. Here, a inductive (contactless) coupling has proven useful. It also leads to a reduction of production cost of the device since bonding and a socket are no longer necessary. A capacitive coupling is conceivable as well.

Using the above principles, SAW biosensors have been developed and investigated by us. Using $LiTaO_3$ as the substrate material, delay lines with an operating frequency of about 250 MHz and a delay of about 2 µs were produced in a one- or two-port (e.g. for the use in oscillator circuits) configuration. With one-port devices, phase sensitive detection must be used as the measurement technique.

Like for all acoustic sensors, it is important to account for the influence of temperature onto the SAW velocity. This can be done by thermostating the device by, e.g., a Peltier element, and additionally, by compensating for the most part the temperature effect by a second reference track on the same device. Compensation is associated with a rather high expense, and at a duration of our measurements of about one hour, it has turned out to be unnecessary.

Supply of the sample liquid can be done by pipetting or in a flow-through cell. Which technique is to be preferred depends on the demands of the user. The pippetting technique requires that care is taken to avoid evaporation of the sample liquid by a suitable measuring cell, otherwise the associated cooling of the sample and the loss of liquid can lead to erroneous results. Inductively coupled sensor devices are especially suited for flow-through cells since they can easily be tightened against the outflow of liquid.

The measurement technique preferred by us is the phase measurement, since it is insensitive to the large changes of SAW velocity and attenuation when the sample liquid is applied, and because the expense for the necessary electronics is only slightly higher than for an oscillator circuit. It also has the advantage that crosstalk and triple transit echoes can be suppressed, permitting the use of simple and more compact one-port devices.

Laser-acoustics for testing thin films and material surfaces

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Surface engineering and thin film technologies are important for the technological development today. Controlling the quality of thin films and surfaces quickly and reliably is essential for cost-efficient technological developments and process controlling.

Whereas varying highly-developed analytical methods are able to detect single atomic layers with high sensitivity and lateral resolution, a limited number of methods such as indentation test, membrane-bending test, or scratch test are available to evaluate important mechanical parameters as elastic modulus, hardness or adhesion with however limited sensitivity for very shallow surface regions.

Surface acoustic waves are a new complementary test method that is non-destructive and enables elastic modulus, density or thickness to be evaluated for films down to some nano-meters.

This acoustic wave mode is employed by the laser-acoustic test method described in the contribution. Short laser pulses of 0.5 ns duration are used to generate a wide-band spectrum of surface acoustic waves. The low frequency waves penetrate deep into the material and therefore propagate with the velocity determined by the substrate. Increasing the frequency reduces the penetration depth of the surface acoustic waves and makes them more sensitive to the film, accompanying with the phase velocity approaching to velocity of the film material. Consequently, the propagation velocity of surface acoustic waves propagating in coated materials depends on frequency, termed dispersion. The dispersion depends on the combination of film and substrate material and the film thickness as well. The laser-acoustic method measures the phase velocity of the surface acoustic waves, termed dispersion curve. In the second step, a theory for the dispersion of the surface acoustic wave in coated materials is used to deduce the interesting film parameters. This procedure represents the inverse solution of the dispersion relation that must be performed numerically.

The laser-acoustic technique has turned out to be useful for testing, for example, super-hard materials, ultra-thin films and damage layers. The ability of testing comparably rough material surfaces (average surface finish $R_a \leq 1\ \mu m$) has enabled the damage depth of semiconductor wafers after the sawing process to be determined or the Young's modulus of CVD-diamond coatings on cutting tools to be measured, indicating the diamond-likeness of the coating.

The advantages of the method have been demonstrated for some practical examples:

- super-hard nano-meter films,
- damage layers in semiconductor materials,
- porous low-k films,

For a test series of diamond-like carbon films, the results of the laser-acoustic method have been shown to be in a good agreement with those of the alternative technique of the well-known indentation test.

SAW Sensor Systems for the Measurement of Physical Parameters in Automotive Applications

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SAW physical sensor and identification systems can advantageously be employed in the automotive manufacturing process and for the improvement of the automobile performance, safety or comfort features, e.g. by the wireless measurement of tire parameters, torque measurements in the power train or on the steering column for electric power assisted steering systems. SAW based viscosity measurements can deliver information about quality of engine oil, fuel, brake or battery liquid. Three different examples (identification system, intelligent tire, oil quality measurement) out of a manifold of possible applications are chosen to present the basics of wireless and wired SAW sensor systems and to explain system specific properties. By means of a SAW identification system, which allows a highly flexible assembly and the usage of only a single ID system in the entire production process different coding schemes, approaches for economic coding, reader architectures and performance estimations are presented. A tire which provides information about its current state will considerably improve drivers' safety as well as vehicle stability. Thus it is common knowledge that 80 percent of all flat tires result from a gradual loss of tire pressure. If such a creeping decrease in tire pressure can be detected in time, many accidents could be prevented. Several battery-powered systems have hitherto been implemented for the measurement of tire pressure. However, they suffer from a limited operating life of the pressure sensor unit due to the extreme tire environment. This problem can be circumvented with passive wireless SAW pressure sensors. Measurements of road friction with SAW sensors directly vulcanized into the tread elements of tires supply valuable information for future driver-assistance systems and for optimizing slip-control and vehicle-stability systems. For an even further improvement of engine performance, an early detection of possible engine defects and an extended service interval oil quality is an important parameter. Using SAW devices for oil-viscosity measurements on-board diagnosis of oil condition can be carried out in an elegant way.

Ultrasonic sensing using volume and guided waves

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We demonstrate high-resolution topographic imaging by means of Phase-Sensitive Acoustic Microscopy (PSAM) in air at 1.3 MHz. This results in an axial resolution of 1 µm. A typical application in fingerprint recognition is shown. As another example of ultrasonic sensing transmission PSAM of functionally-graded materials is discussed. From the time of flight of longitudinal and transversal volume waves in the sample local mechanical properties are derived. Maps of Poisson ratio, Young’s modulus and shear modulus are shown. In certain cases volume waves turn out to be inappropriate for ultrasonic testing. One of these cases is the directional solidification of metals, where the detection of the solid-liquid interface is desired. The control of the position and velocity of the solid-liquid interface is decisive for the resulting morphology of the metal. Scattering at grain boundaries, however, often makes the detection of well-defined echo signals impossible in the case of volume waves, and only the use of low frequency guided waves results in a good signal-to-noise ratio. Using short pulses of axial-radial guided waves the directional solidification of gallium as a model system, as well as of an aluminium-magnesium alloy is investigated. Moreover, the homogeneity of the samples before and after melting is characterised by the same method using an electromagnetic transducer (EMAT) for contactless excitation of axial-radial guided waves at variable positions along the cylindrical metallic sample.

Fundamentals of non-linear underwater acoustic systems

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The use of non-linear transmitting for underwater acoustical applications offers new possibilities to solve recent problems of tasks for sub-bottom investigations.

At very high sound pressure the density of water and therefore the sound velocity is changing non-linear. The transmitted sound waves will be distorted and the spectrum of the transmitted signal will be changed.

To use this non-linear effect in practice there can be transmitted two slightly different frequencies with high sound pressures at the same time. Due to the non-linear interactions of the transmitted primary frequency signals beneath the transducer new secondary frequencies arise in the water. One of them is the difference frequency which is a low frequency that penetrates the bottom and can be used to receive detailed information about sediment layers or embedded objects beneath the bottom surface

The main advantages are the small beam width valid for the primary and secondary frequencies and the high system band width which allows to transmit very short, low frequency signals with small transducers. Further the directivities of the secondary frequencies have no side-lobes. These properties more than compensate the disadvantages of the high necessary transmitting power and the low electro-acoustical efficiency for the difference frequency.

The parametric sound field is formed within the interaction length in front of the transducer. In contrast to the directivity of the primary signals there are no local minima or maxima in the near-field area for the difference frequency. Therefore non-linear echo sounders can be used in very shallow water. Due to the interaction length of the parametric transmitter, the decrease of the sound caused by geometrical losses and physical attenuation will be lower compared to linear echo sounders. The interaction length depends on the transducer dimensions, the wave length of the primary frequencies, the coefficient of non-linearity and the sound pressure.

The sound pressure of the primary signals and the ratio between primary and secondary frequencies mainly determine the efficiency for the difference frequency.

The difference frequency is able to penetrate the sea bottom and therefore used for receiving of sub-bottom information with an excellent horizontal and vertical resolution as never seen before. It becomes possible to transmit very short pulses with a small beam width – despite of a small active sound area of the transducer. A new generation of mobile sub-bottom profilers is using this technology now.