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Q-SENSE AB

QCM-D — Bengt Kasemo charts the progress.

Bengt Kasemo has an international reputation as a researcher in surface science, with ten patents and between two to three hundred scientific papers to his credit. He is one of the key figures behind the setting up of Q-Sense and the application of the QCM-D (Quartz Crystal Microbalance with Dissipation monitoring) technique in research applications.

Today, he holds the position of Professor of Chemical Physics within the Institution of Applied Physics at the Chalmers Technical University in Gothenburg, Sweden, where he leads a group of around 35 people.

Broad potential for QCM-D

His interest in quartz crystal measurement goes back 25 years, at a time when its use was limited primarily to vacuum environments. Then, the technique was generally used for thin film deposition monitoring, while in Kasemo's group its use was focused on studies of various surface processes such as metal oxidation, metal hydride kinetics, gas adsorption kinetics and catalytic reactions. Thanks to key developments by Anatol Krozer, Michael Rodahl and Fredrik Höök, who worked with Kasemo as graduate students on various projects, the QCM technique was broadened to include operation in liquids and, crucially, also allowed measurement of the dissipation (D) factor that gives precise details of viscoelastic properties of thin films. "This added a new dimension to the QCM, which normally measures only the mass uptake on the sensor surface," explains Kasemo.

Indeed, the ability of QCM-D instrumentation to measure both the frequency-related mass uptake and the D factor is not merely adding a new measured quantity, points out Kasemo. Combined with theoretical work using algorithms developed by Prof. M. Voinova and others, it revolutionises the possibilities to extract a wide range of quantitative information, for example, about biofilms and biomolecules on surfaces such as those required for the development of new biomedical diagnostic tools.

By 1995 it became clear that the modified measurement system, now dubbed QCM-D, had wide scientific application and it was decided to seek venture capital to set up a commercial company. The business idea was made a study project at the Chalmers School of Entrepreneurship, where Krozer, Rodahl and Höök managed to put together a successful proposal culminating in the launch of Q-Sense in 1996. Kasemo is excited about the opportunities for the technique in a whole range of scientific research. "I believe the potential is very high, considering the rapid development of the biomedical and biotech areas, with many possible spin off effects," he says. "In addition, there are a number of extremely interesting applications in other

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Welcome to the first edition of Q-News!

Word of mouth has contributed greatly to the growth of Q-Sense over the past couple of years. As the potential of the QCM-D technique is becoming apparent we feel it is time to begin to share the excitement of this new approach with the wider community. Hence the arrival of Q-News as a means of communicating our progress.

Thin film technology is a technology that is attracting significant interest in a number of scientific areas. While researchers have the techniques to create and build thin films, they also need increasingly sophisticated tools that can help them understand the behaviour and structure of the microscopically thin layers they produce.

The QCM-D is such an advanced technique. Importantly, it has entered the commercial area and is being used by prominent researchers in a number of key scientific fields including paper chemistry, biomaterials, biofouling and surfactants. The technique is promising because it has unique properties that offer researchers a new way to study complex surface related chemical reactions or biological activity.

Q-News has been designed to bring you the latest information and news about the development and application of this maturing technology. In our first issue we bring you an interview with Bengt Kasemo, one of the founders of the company and a key figure in the development of the technique. Plus, we have a report on a major new application of the measurement technique at the Institut Charles Sadron in Strasbourg where Prof Gero Decher is using QCM-D in the study of polyelectrolytes.

Happy reading!

Eva-Carin Tengberg
Managing Director

qsense®

areas, such as polymer technology and information technology hardware. It is a real challenge to choose and focus on a few of them."

Lessons learnt

From his academic position, Kasemo has watched the transformation of QCM-D from a laboratory tool to a marketable product. "It is an exciting – and sometimes frustrating lesson," he notes. Those involved with the project have learnt a number of lessons in moving the QCM-D technique from a university to a commercial environment, he believes.

"I think it has now been demonstrated that there is a market need for this technique and this specific instrument," says Kasemo. "The company still needs to demonstrate the usefulness of the technique to several market segments that have not discovered its potential yet." That said, Kasemo sees his current role in relation to Q-Sense as helping to support the company's development whilst remaining firmly in the academic arena where his skills are best exploited. He feels that his main contribution in this regard will be to continue using the QCM-D in research. Also, Kasemo is convinced that collaboration with users is a key in the future of the company as a whole. "Feedback from customers and the ability to listen to their needs is a key factor for success," he points out.

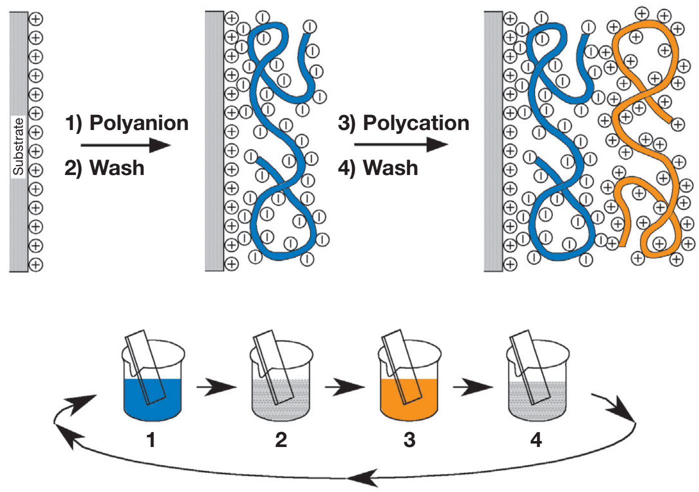
Well-placed to give feedback

As a researcher, Kasemo is well placed to give such feedback. As QCM-D has evolved, so has the nature of the work carried out by his group at Chalmers. The group's research is very diversified, though there is a common denominator in that everything revolves around surfaces and surface processes. Increasingly, the research focus has moved from gas-surface interactions related to studies in fields such as catalysis to those concentrating on biointerface studies which is where Q-Sense sees a tremendous growth potential. This is an area that is changing very rapidly with new demands and opportunities opening up virtually each day. The distance from basic research to commercial reality is very short.

Getting Layered up.

Professor Gero Decher at the Institut Charles Sadron, Université Louis Pasteur, Strasbourg in France works in the field of polyelectrolyte multilayers, an area he started about ten years ago. Today his work is generating tremendous scientific and commercial interest because it offers the ability to make extremely thin layers using a potentially low cost manufacturing technique.

The application of these thin sandwich layers is equally large and includes electroluminscent, non-linear optical and electro-optical electronic devices, biosensors, biocompatibilisation of surfaces for medical use, separation membranes, catalysis, ultra-thin batteries and tiny chemical reactors.



The process of laying down polyelectrolyte layers.

Focus on polyelectrolyte multilayers

Decher's group has its main focus on multi-material surface layers. These are studied in Strasbourg with research projects in both fundamental and applied science. This layer-by-layer assembly, LBL, technique can create multilayer composite films from a variety of materials with nanoscale precision. At Strasbourg, the team mostly uses electrostatic attraction to attach molecules onto solid surfaces.

Multilayer films created by the LBL technique should not be confused with so-called LB films. LB films were named after I. Langmuir and K.B. Blodgett, who originally developed their technique some 65 years ago. Since then, the LB technique has become a very elegant tool in many laboratories, but has never been used successfully in a commercial application. The idea for making LB films is quite intriguing. It involves forming a film of molecules on the surface of water and then transferring this film onto a solid surface when it is dipped into the water. This has major drawbacks, however, because the process requires special equipment and only works with a limited range of molecules.

The technique developed by Decher is based on adsorption from solution, which is simpler and less expensive than the Langmuir-Blodgett approach. Importantly, LBL assembly can be applied to a broad range of materials and surfaces.

Charged up

In essence, Decher can form thin films from virtually any charged polymer. This is done by simply immersing the carrier material, such as glass, or even metal or plastics, in a solution of the desired polymer. If the original surface charge of the carrier substrate is too small, its surface may need additional treatment such as exposure to a plasma or chemical activation. In the case of glass, the surface is already negative and the first layer can be directly deposited. Since the polycation contains hundreds of positive charges along its chain, it is attracted and sticks to the negatively charged glass surface. This creates the first layer of the sandwich and takes only a few minutes, depending slightly on the material involved.

Once a thin layer of positively charged molecules covers the surface, further adhesion is halted as the charged layer repels further polycations. After rinsing the substrate and its new positive layer in water, the same procedure can be carried out to add a negative layer to build up a sandwich that is as complex as required. As long as the polyion charge changes from layer to layer, more than 1500 consecutive layers can be deposited.

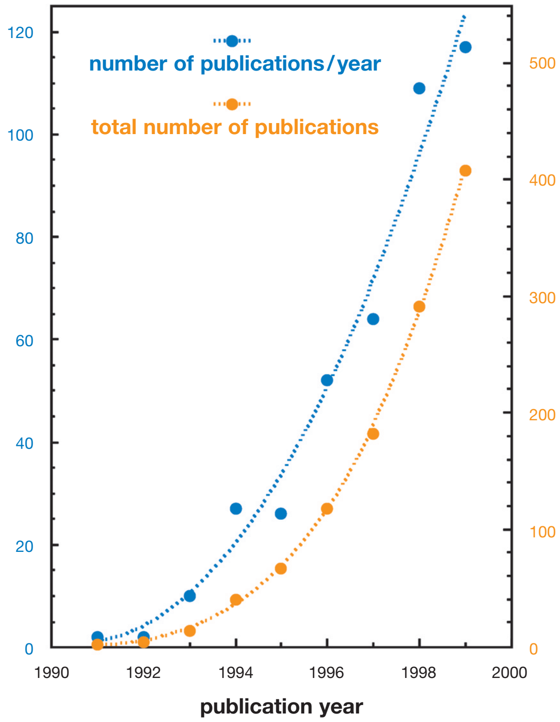
Decher has built layers on substrates of silica, glass, quartz, diamond, metal and polystyrene. "Our technique allows us to make surface modifications while maintaining molecular control, to create multi-material hybrid thin films," points out Decher. "We can put polymers, proteins, DNA, colloids onto a variety of surfaces." It is ideally suited for industrial use as it does not require the application of harmful solvents.

Industry has already caught on and the first devices with LBL films are expected to be on the market this year.

Unique tool

At the frontier of this type of research, the ability to make layers is not sufficient. Researchers need to understand what is happening at these interfaces and during the layer formation. Properties of the multilayers are influenced by the deposition process, the environment used for deposition as well as the polyelectrolyte types and their interactions. In addition, information is needed relating to the way in which layers build up, and the changes to layers over time induced by the environment. A variety of traditional measurement techniques including spectroscopy, electron microscopy or X-rays are used to determine the nature and behaviour of multilayers alongside QCM-D. For Decher, QCM-D is a tool which can help provide a more detailed physical understanding of the deposition process and of the resulting multilayer process. Research is underway to explain the dependence of the structural details of multi-component films on the exact chemical nature of the components.

Decher says that QCM-D is "uniquely suited to follow the adsorption of molecules and objects on surfaces and to characterise adsorption kinetics under a variety of experimental conditions. We benefit enormously from the D option (which relates to viscoelastic properties) for characterising our films". In regards to this, Ralf Richter, applications specialist at Q-Sense, explains: "We had to apply a special model for the Decher work. QCM-D is applied to layers in the region up to 50 nm thickness while Decher produces films of around 100nm and more. So, the QCM-D response is different." The model software is called Q Tools.

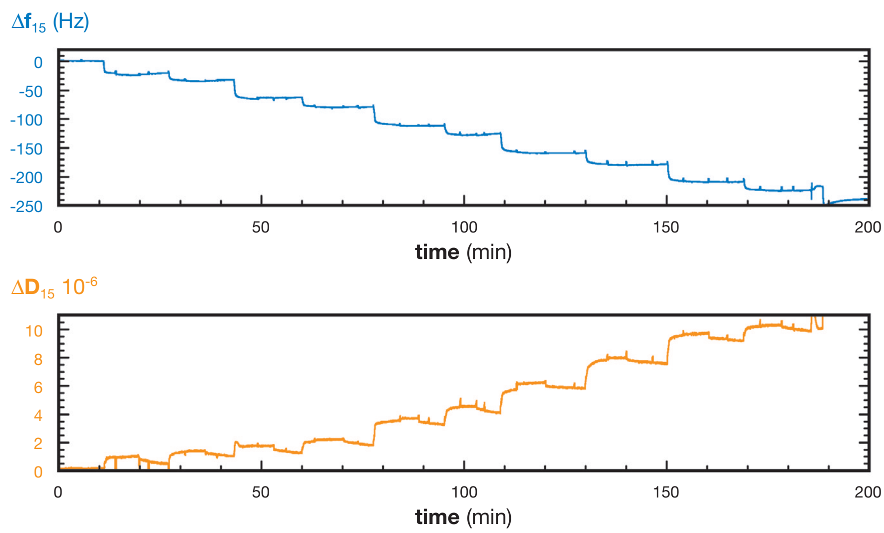


The growth of interest in polyelectrolytes has been dramatic with the increase in paper published in the last decade.

Source: P. Bertrand, A. Jonas, A. Laschewsky and R. Legras, Macromol. Rapid. Commun. 21 (2000), 319-348

"The QCM-D technique allows structural changes to be followed in real time and it has the ability to obtain information on water content," points out Richter. This is particularly useful for layers incorporating biomolecules such as proteins, an area in which Decher is also involved.

Having another measurement tool is extremely helpful when it comes to exploring such a novel field. "Our technique is so new that many things need to be investigated," concludes Decher. "More than 40 independent research groups have already joined our efforts."



QCM-D allows researchers to measure the buildup of polyelectrolyte multilayers and their structural changes in real-time. This example shows the deposition of alternating positive and negatively charged polyelectrolytes. The measurement was conducted by Gero Decher's research group.

Expertise on tap.

Q-Sense is working with a number of prominent academics to further identify and develop the QCM-D technique in research applications. These researchers are already well-versed in the technology and application of quartz crystal measurements and form part of a network of Reference Centres. This network is not only intended to be a valuable resource of expertise to the company but can also provide support and information to new users of the technique.

For example, today the QCM-D technique is used to characterise interactions between macromolecules and living and solid surfaces. Much work has also been done on the investigation of various biomolecule-surface interactions such as protein adsorption and phospholipid bilayer formation, bacteria and cell adhesion. Eva-Carin Tengberg, Q-Sense managing director, points out, “We hope that the close connection between different reference centres will have strong synergetic effects. On the one hand, it will keep the research groups updated with recent development of the QCM-D technique and on the other keep Q-Sense updated of scientific trends and possible new areas of interest.”

Ten centres at present

At present there are ten research groups classed as reference centres based in Sweden, France, Finland, Denmark, Belgium, Switzerland and the US. These centres are based at; The Institut Charles Sadron, Université Louis Pasteur, Strasbourg, France – The Department of Applied Physics, Chalmers University of Technology, Göteborg, Sweden – The Laboratory of Surface Science and Technology, ETH, Zürich, Switzerland – Helsinki University, Department of Forest Products Technology, Finland – Harvard University, Department of Chemistry and Chemical Biology – Stanford University, CPIMA, Centre on Polymer Interfaces and Macromolecular Assemblies – Imec mcp-div., Leuven, Belgium – University of Bordeaux, France – Aalborg University, Department of Life Science, Denmark – Uppsala University, Clinical Immunology, Sweden.

Their research ranges from the study of polyelectrolytes and multilayers, ink adsorption and model surfaces of cellulose, mathematical modeling on material properties of thin films, to studies on biomaterials and creation of specific surfaces.

Making Sense of QCM-D.

Quartz crystal measurement has long been a valuable research tool. Its main use has been in the measurement of mass uptake on a sensor surface. The technique's refinement to create QCM-D provides new opportunities to understand the properties and processes of thin films.

The QCM-D principle employs a sensor that consists of a thin crystalline quartz disc sandwiched between two electrodes. Small oscillations in the sensor at resonant frequencies are created by applying an AC voltage across the electrodes. The particular resonant frequency of the disc is dependant on the overall oscillating mass. The basic equation related to this is $\Delta f = -k \cdot \Delta m$, where k is the mass sensitivity constant.

The sensor can be pre-coated with a huge range of thin films from metals, polymers, ceramics to biomolecules, bacterial and living cells. The addition of the film alters the resonant frequency of the sensor. This frequency change is directly proportional to the film's mass, if the adsorbed film is rigid. For soft or viscous films, the Sauerbrey relation is not valid since the outer region of the film does not follow the sensor's shear motion. Here, measurement of Dissipation (defined as lost energy per oscillation divided by total energy stored in system) is crucial. D (Dissipation) factor measurement of the oscillation allows accurate thickness estimations and viscoelastic properties of floppy films such as:

- Polymer multilayers
- Cell and bacteria adhered to functionalised surfaces
- Elongated proteins or polymers with no specific structure adsorbed to solid surfaces

Importantly QCM-D measurements can be carried out in real time. This is useful in tracking material build up on the sensor's surface such as during protein adsorption.

In practice, a sealed chamber provides a controlled environment for measurements. PC control allows continuous data acquisition and tailored presentation of the final results.

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