Online monitoring of polymer coating process using Sciospec ISX-3 Impedance Analyzer

Online monitoring of deposition or coating processes is highly relevant in a variety of fields including biocompatible coatings for medical implants. As of now mostly "simple" resonance tracking of quartz crystal microbalances is used for these purposes. Currently ongoing joint research by AG Mayr, Leibniz Institute of Surface Modification Leipzig[1] and Sciospec is aiming for more specific insights into the process itself. Especially nano-structural formation and adhesion parameters are of high interest. This application note describes the first step of data acquisition and parameter estimation.

Setup

A high vacuum chamber, with a base pressure of 10^{-7} mbar, was employed to deposited Poly(Bisphenol-A-carbonate) via thermal evaporation.

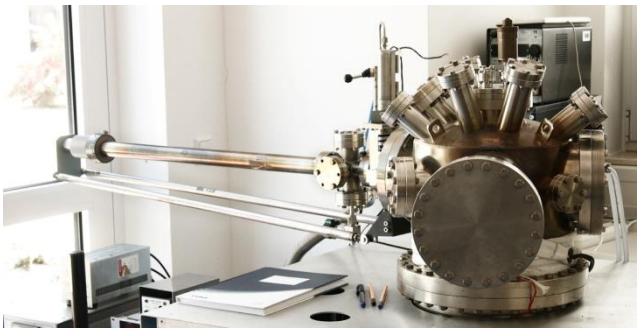

Fig 1: High vacuum chamber

The vapor has been deposited onto a gold coated quartz crystal microbalance sensor (QCM). The 0.55 inch in diameter QCM sensor chip was analyzed by a standard Sciospec ISX-3 impedance spectroscope. The nominal frequency of the sensor is about 6 MHz; therefore the range was set to 5.9 MHz to 6.5 MHz with 2001 points[2]. This very high resolution is needed, since the change in frequency is expected to be just a few hundred parts per million. Since the oscillation frequency of the oscillator sensor is highly temperature dependent, external cooling was applied to hold the temperature of the chip constantly at 18°C with a tolerance of 0.2 K. The quartz crystal chip was attached by a standard sensor head for high vacuum usage with BNC feed through which was connected to the Sciospec ISX-3. For best results all grounding of the measurement and deposition system was connected to one potential.

Measured data

All the data has been displayed and stored with the Sciospec Impedance Analyzer Suite. The resonance

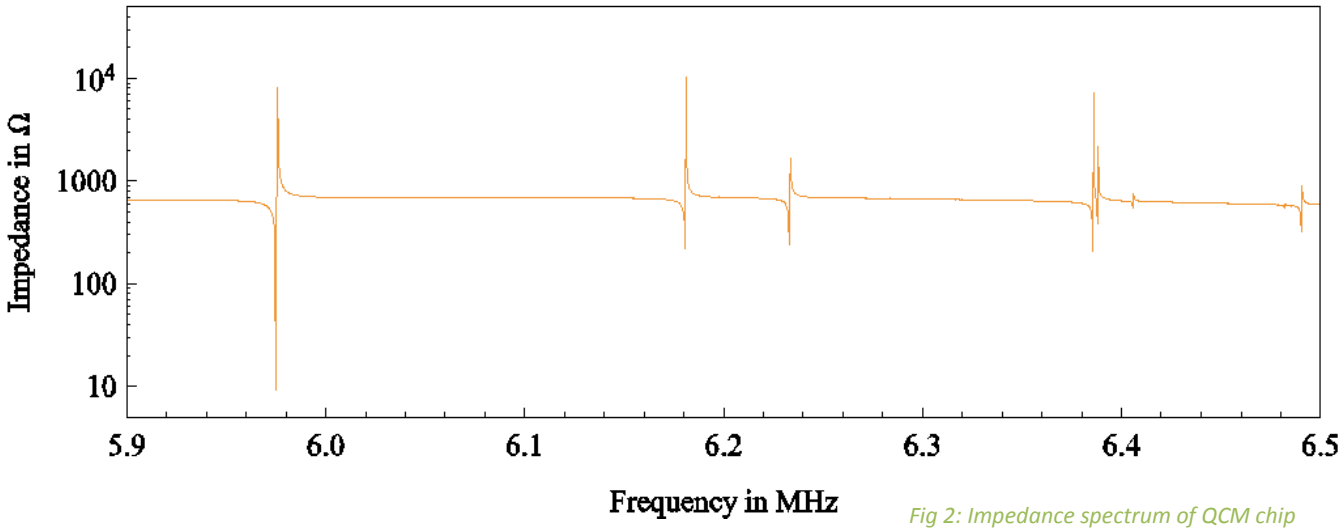

Fig 2: Impedance spectrum of QCM chip

Sciospec Scientific Instruments GmbH
Grimmaische Straße 92
D-04828 Pausitz
GERMANY
Tel: +49 34383 6315 40
Fax: +49 34383 6315 45
Email: info@sciospec.de
www.sciospec.de

frequency was tracked by peak fitting for each spectrum (Fig 2). The resulting frequency change was translated into the thickness (given in nanometers [nm]) of the deposited PBA (Fig 3) by the well known "Sauerbrey"- equation[3]. This allows for a very sensitive in situ monitoring of the deposition process, which has been proven to be a useful approach in this kind of setting.

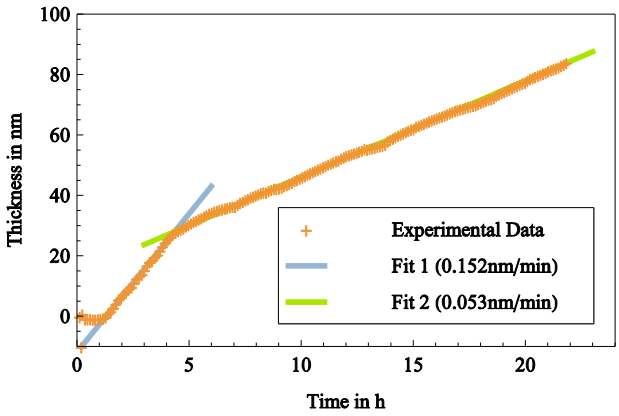

Fig 3: Thickness of the deposited Poly(Bisphenol-A-carbonat) in nm over time in hours

Evaluation

The data divides into three intervals. The first corresponds to the heating of the PBA. The evaporation temperature of about 230°C is reached after approximately one hour. This interval shows no relevant change in the spectra since no material is deposited. The temperature is still rising to 300°C at one and a half hour and then held constant over the complete process. After about one hour the temperature is therefore high enough so the deposition started at a rate of about 0.152 nm per minute. This seems to correspond to the formation of unconnected isles of PBA on the gold surface of the quartz crystal. The third interval starting at about four and a half hours after the beginning is not yet fully understood. The deposition rate abruptly changes to about one third (0.053 nm per minute) of the initial rate. This coincides with the formation of one single layer of the deposited material. Therefore a connection of the islands of the material seems to be a good possibility. Closer studies in combination with atomic force microscopy (AFM), best in situ, will be needed for a better understanding of this formation process.
The final thickness of 84 nm has also been rechecked by X-ray reflectivity measurement to be correct in the range of one nm.

Outlook

Closer analysis of the secondary modes of the impedance spectrum have revealed clues to the nano-structrural formation within the coating process. Further investigation will lead to a closer understanding of the adhesion of the PBA molecules to the gold surface. As mentioned above, a study including AFM measurements in combination with even higher resolution impedance spectroscopy on resonant high-Q systems will to be the next step. Sciospec in cooperation with the AG Mayr[1] is developing a fully integrated quartz crystal microbalance sensor with automatic temperature control for the use in ultra-high vacuum applications. The sensor will attach seamlessly to the existing Sciospec products and will be integrated in the Sciospec control software for easy to use setup.
In this experiment the Sciospec ISX-3 system proofed to be an effective measurement tool for online in-process monitoring in coating applications.

This application note is part of the results of the joint research between Sciospec and the Leibniz Institute for Surface Modification (IOM) Leipzig. The project "***Innovatives Verfahren zur mechanischen Charakterisierung von Grenzflächen für die Entwicklung neuartiger biokompatibler Oberflächenbeschichtungen*** " is funded by EFRE and the Free State of Saxony with support of the SAB.

[1]Leibniz-Institut für Oberflächenmodifizierung e. V., Permoserstr. 15, D-04318 Leipzig
[2]Datasheet Sciospec ISX-3, www.sciospec.com
[3]Sauerbrey, Günter (1959), Zeitschrift für Physik 155 (2): 206–222

Sciospec Scientific Instruments GmbH
Grimmaische Straße 92
D-04828 Pausitz
GERMANY
Tel: +49 34383 6315 40
Fax: +49 34383 6315 45
Email: info@sciospec.de
www.sciospec.de