Application Note

# Detection of self-assembled monolayers (SAMs) using contact angle measurements

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*Surface treatments like the atomic layer deposition can be utilised to encapsulate medical implants with a nanometre-thin metal-oxide-coating, protecting implants from the harsh environment of the body. However, atomic layer deposition also encapsulates active surface areas of the implant, such as electrodes made from platinum, which negatively affects the conductivity of those electrodes. Thus, it is necessary to exclude active surface areas like electrodes from the surface coating process. Self-assembled monolayers (SAMs) can be used to passivate surface areas which should not be coated. SAMs adsorb exclusively on the active platinum surface areas and, due to chemically inert end groups, prevent the oxide-coating from growing on those areas. In this experiment, SAMs out of a vapour phase and out of a solution were applied to platinum surfaces. Using the optical contact angle meter OCA 200 and the PDDS picolitre dosing system from DataPhysics Instruments, differently treated surfaces were examined and quantified using contact angle measurements.*

**Keywords: thin metal-oxide-coating, atomic layer deposition (ALD), self-assembled monolayers (SAMs), medicinal implants, platinum, electrodes, contact angle measurement**

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## Introduction: Encapsulation of medical implants using the ALD-process

Active implants, such as retinal implants or neural implants, are in direct contact with the harsh environment of the body after implantation. Elevated temperatures, physiological salt solutions and foreign body reactions put stress on the implant. Therefore, it must be protected with a suitable encapsulation strategy. Atomic layer deposition (ALD) is a cyclic coating process, which can be used to apply ultra-thin, conformal and defect-free layers. The coating process is characterised by self-limiting surface reactions, which enables control of the layer thickness even below one nanometre. The control is gained by separate and repeatable steps in the coating process, namely: introducing the layer-forming chemicals, purging, reactivating the surface and purging again. As a result, thin metal-oxide layers can be deposited. Such layers perform as excellent barriers: They protect both the implant from the harsh environment of the body and the body from the implant's potentially harmful materials or electrical currents.

The OCA 200 contact angle meter from DataPhyiscs Instruments was specially developed to measure on the smallest surface areas.

## Objective: Active surface areas should not be coated

Due to growth kinetics, lateral growth control of the coatings is difficult. Nevertheless, active surface areas of the implant, such as its electrodes made of platinum, should not be coated. The electrodes are used for stimulation or the recording of nerve stimuli. An electrically insulating barrier, such as the metal-oxide layer formed through ALD, dampens the biosignals and thus reduces the performance of the implant. By pretreating the active surface areas with so-called self-assembled monolayers (SAMs), the growth of an oxide metal coating on active surface areas can be prevented.

SAMs consist of: a head group, which adsorbs on the substrate; a hydrocarbon body and a functional end group. The functional end group is usually a non-polar, chemically inert compound, such as methyl. It prevents the growth of further layers of coating on its top. The SAMs can be applied to the substrate from the vapour phase or directly from a solution. SAMs adhere only to the active surface areas made out of platinum.

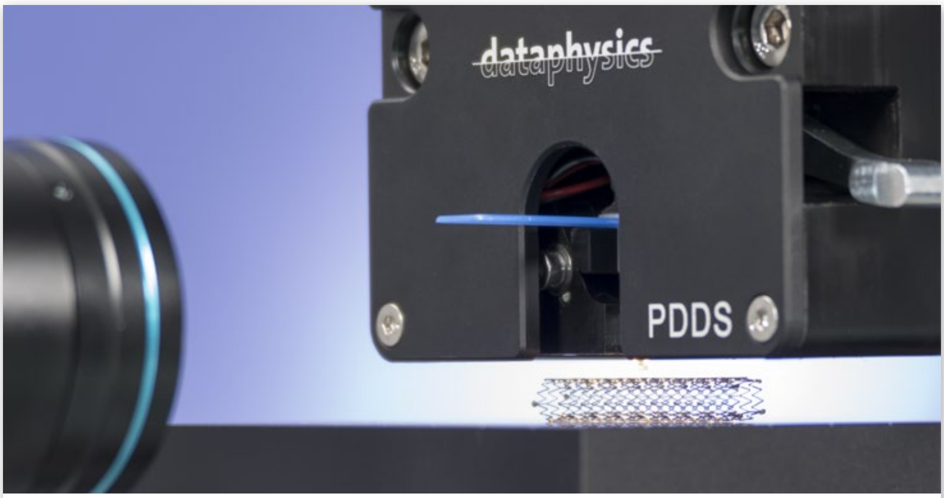
## Experiment: Using contact angle measurements to detect SAMs

With the help of a contact angle measurements, the presence of SAMs on a surface area can be detected quickly and easily. In a recent experiment done at the Department for Mechanical and Medical Engineering at the Furtwangen University, Germany, SAMs from different phases were applied to platinum surfaces.

The starting materials for the samples were silicon wafers. Those were coated and then cut to a size of 15x15 mm². The surface of the first sample consisted of untreated platinum. The second sample was treated with SAMs out of a solution, the third sample with SAMs out of a vapour phase. The SAMs were examined and qualified using the OCA 200 contact angle meter and the accompanying SCA 20 evaluation software from DataPhysics Instruments.

Water droplets were applied to the three sample substrates using a high-precision picolitre dosing system: the PDDS. With the PDDS, it is possible to accurately dispense drops down to a minimum volume of 30 pl. This is particularly advantageous for discretely investigating surface areas with small diameters of hundreds of micrometres, like electrodes on implants.

For this experiment, a programming routine was written, which allowed fully automated drop dispensing and drop shape measurement of 20 equally-sized drops on one the sample surface.




With the PDDS picolitre dosing system, dispensing drops with a minimum volume of 30 pl is possible.

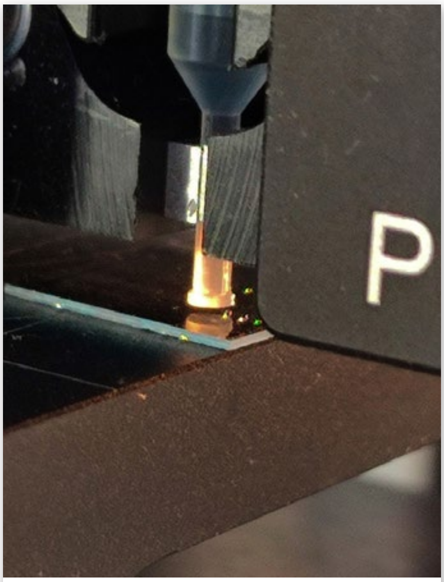
## Results:
## Contact angles enable quantitative evaluation

The images below show water drops on differently pre-treated platinum surfaces. The evaluation software can calculate the contact angle between the water droplet and the substrate by means of contour analysis. As can be seen, the contact angle of the water drop changes depending on the pre-treatment method: The untreated surface of the platinum substrate has a low contact angle of approximately 40°, which means that this surface has a hydrophilic (water-loving) character. The platinum surface treated with SAMs out of a solution has a contact angle of 126°, which indicates a hydrophobic (water-repellent) character. The platinum surface treated with SAMs out of a vapour phase shows a contact angle of about 72°, a value that lies in between the other two values. The platinum surface with SAMs out of a vapour phase can also be described as hydrophilic.

## Discussion:
## SAMs out of a solution are more densely packed

The results of the experiment allow further conclusions about the nature of the SAMs on platinum surfaces. The pictures show that the platinum surface with SAMs out of a solution has a higher contact angle than the surface with SAMs out of a vapour phase. It can be concluded that the SAMs from the solution adsorb tightly packed onto the surface of the platinum substrate. In contrast, the measured contact angle for the SAMs out of a vapour phase suggest that those SAMs are less densely packed.

With the contact angle measurement system from DataPhysics Instruments, it was thus possible to make a quick, qualitative statement about the surface character of the substrates under investigation in this experiment.



The sample table of the OCA 200 contact angle meter can be moved automatically in all directions.

Programming routines allow automatic dosing with syringes (see picture) or the PDDS dosing system.

## Summary:
## Detection of self-assembled monolayers

In this experiment, three platinum surfaces were investigated at Furtwangen University: untreated platinum, platinum with SAMs out of a vapour phase and platinum with SAMs out of a solution. All three samples were wetted with water droplets and the respective contact angles were measured using an OCA 200 contact angle meter from DataPhysics Instruments. The dosing system PDDS made it possible to dose drops with minimum volumes of 30 pl. The precision of the PDDS will later make it easy to measure smallest electrode surface areas on implants.

Through the contact angle measurements, is could be shown that SAMs, through their end groups of methyl, make the platinum surface areas hydrophobic (water-repellent). The results show quantitative differences of the contact angles: the untreated surface has a contact angle of about 40°, the surface with SAMs out of a vapour phase an angle of about 72° and the surface with SAMs out of a solution a contact angle of 126°. This could be due to the fact that the SAMs ouf of a solution adsorb very densely on the surface, while the SAMs from the vapour phase seem to be less densely packed.

A contact angle of approximately 40° is formed on untreated platinum (left). The platinum surface coated with SAMs out of a solution forms a contact angle of 126° (centre). The platinum surface coated with SAMs out of the vapour phase shows an angle of 72° (right).

# Optical contact angle measuring and contour analysis system for microscopic and macroscopic structures

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The OCA 200 is the contact angle measuring and drop contour analysis system for microscopic and macroscopic surface structures.

The software-controlled, electrically-driven optic alignment enables the OCA 200 to adjust the observation angle and to automatically focus on a drop of liquid.

With the trendsetting 10-fold zoom lens and the reliable autofocus-system by DataPhysics Instruments, the OCA 200 is equipped to handle samples like macroscopic silicon wafers as well as the microscopic mesh structures of coronary stents. In combination with the high-performance camera, even smallest drops of highly volatile liquids can be monitored.

The electrically-driven sample table makes it possible to position micro-structured samples with highest precision and exceptional speed for quick, automated measuring procedures.

With an electronic multiple direct-dosing system DDE/x and up to four electronic syringe modules ESr-N, liquids can be deposited on a macroscopic sample and its surface parameters can be determined automatically.

For the analysis of micro-structured samples, the nanolitre dosing system is available, which can generate small droplets down to a minimum volume of 10 nanolitres. These droplets are small enough to fit, for example, between the screw threads of a dental implant.

Even smaller structures can be analysed using the PDDS picolitre dosing system, which can dose smallest droplets with a minimum volume of 30 picolitres. Such drops allow contact angle measurement, for example, on an individual wire in the mesh structure of a coronary stent or on single fibres.

## Features of the OCA 200:

- Sample table adjustable in all three directions in space with electronic high-performance axes
- Video-measuring-system with high-performance camera with up to 3250 frames/s and USB 3-interface
- Powerful 10-fold zoom lens
- Software-controlled, electrically-driven optics alignment for autofocus and adjustment of the observation angle
- LED-lighting with manual and software-controlled intensity including automatic temperature drift compensation
- TP 50 control panel with touch screen and precision control wheel
- Compatible with manual direct dosing systems with one or two syringe modules
- Compatible with the electronic multiple direct dosing systems DDE/x, with up to four syringe modules
- Compatible with the nanolitre dosing system
- Compatible with the PDDS picolitre dosing system
- Compatible with different environmental chambers, for measuring at temperatures ranging from -30 °C to 700 °C
- Compatible with a humidity generator of the HGC-series

**We will find a tailor-made solution for your surface science use case and will be pleased to provide you with an obligation-free quotation for the system that fits your needs. For more information please contact us.**

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