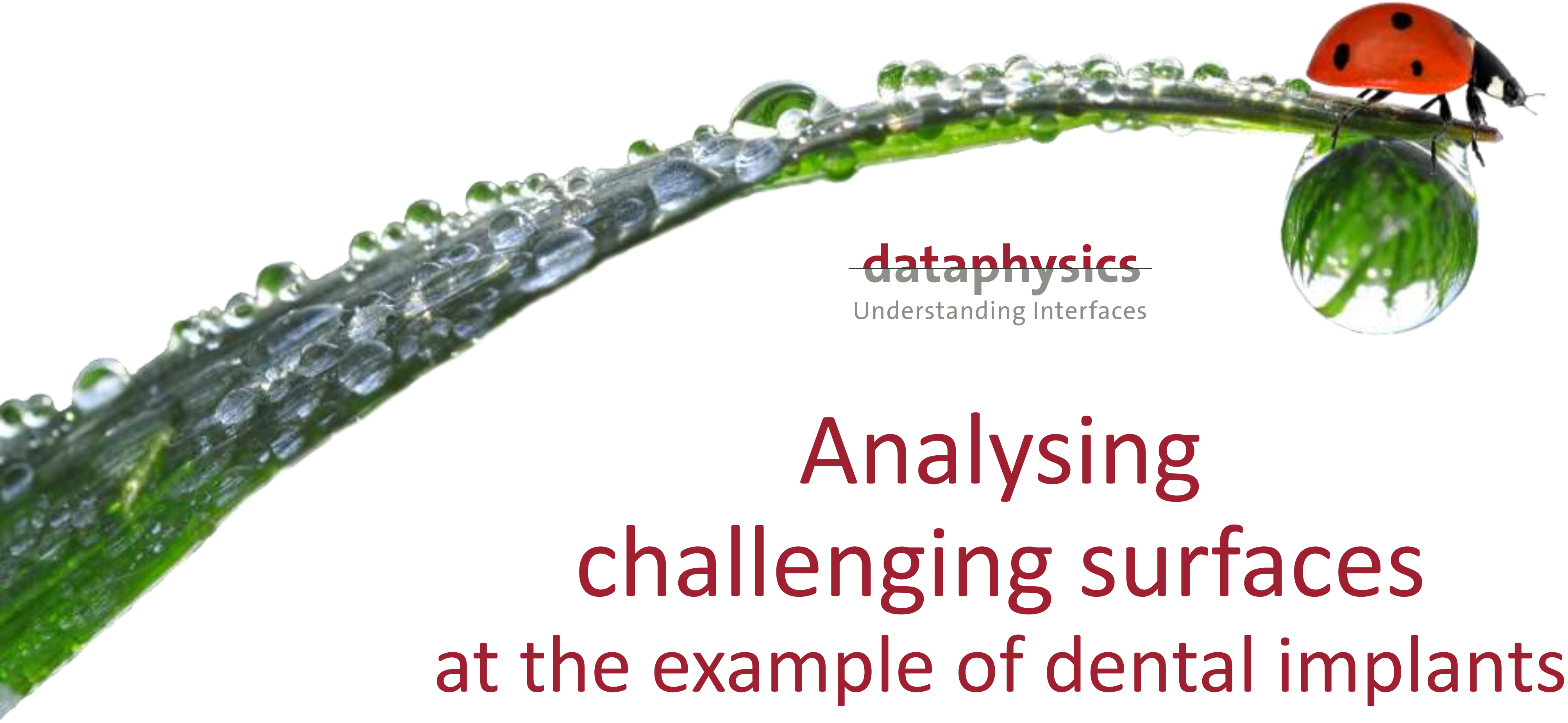

dataphysics
Understanding Interfaces

Analysing challenging surfaces
at the example of dental implants

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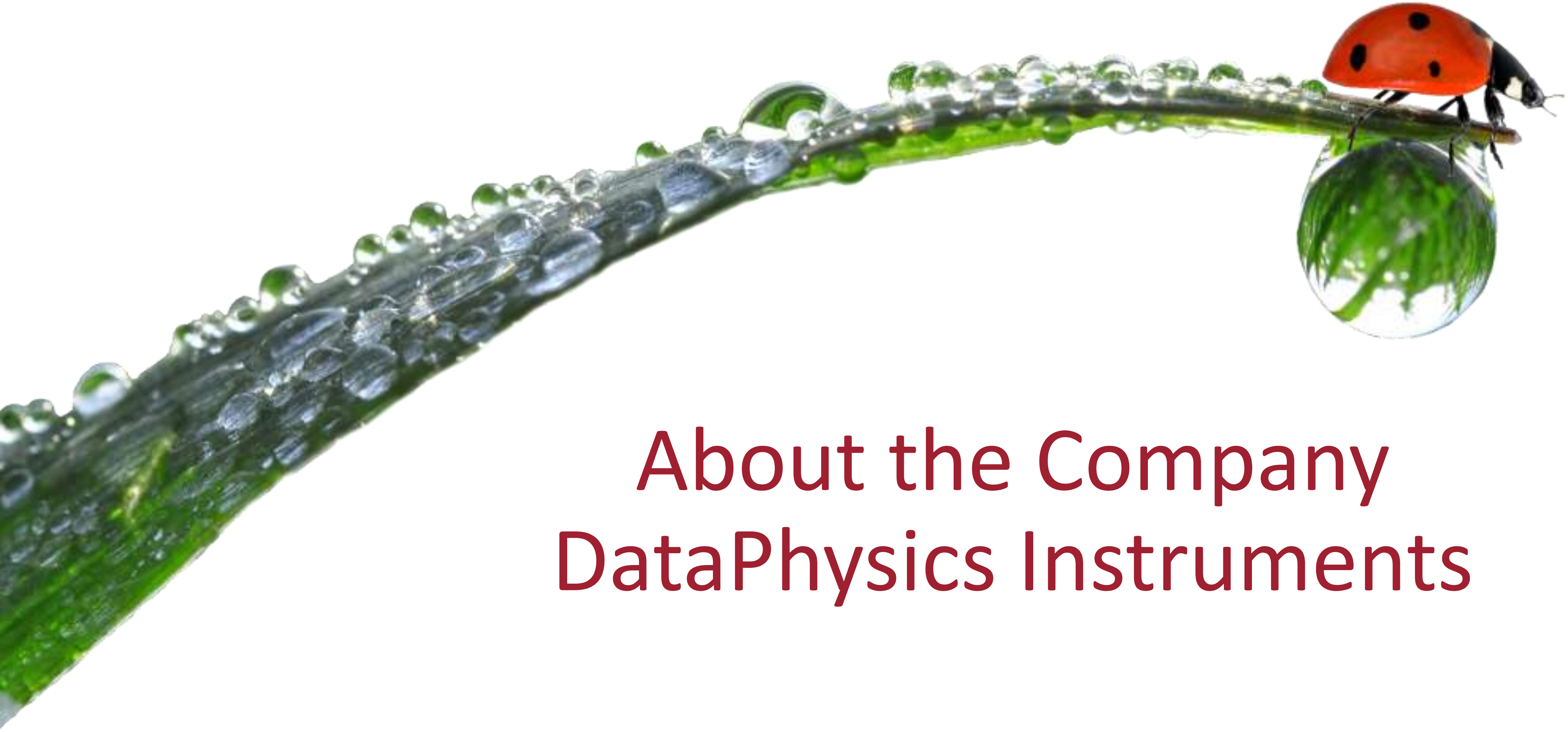


About the Company DataPhysics Instruments

About DataPhysics Instruments

- German manufacturer of high-quality laboratory measurement devices to characterise interfaces and surfaces
- our unique selling points:
  - manufacturing affordable standard solutions as well as bespoke devices for the most challenging experimental tasks
  - versatile modular device systems solving all measurement challenges
  - developing intuitive software for all our devices
  - worldwide distribution network
  - over 25 years of experience in surface science applications

Company Product Portfolio

OCA Contact Angle Meter

DCAT Force-based Tensiometer

Company Product Portfolio

ZPA 20 Zeta Potential Analyzer

MS 20 Dispersion Stability Analysis System

Company Product Portfolio

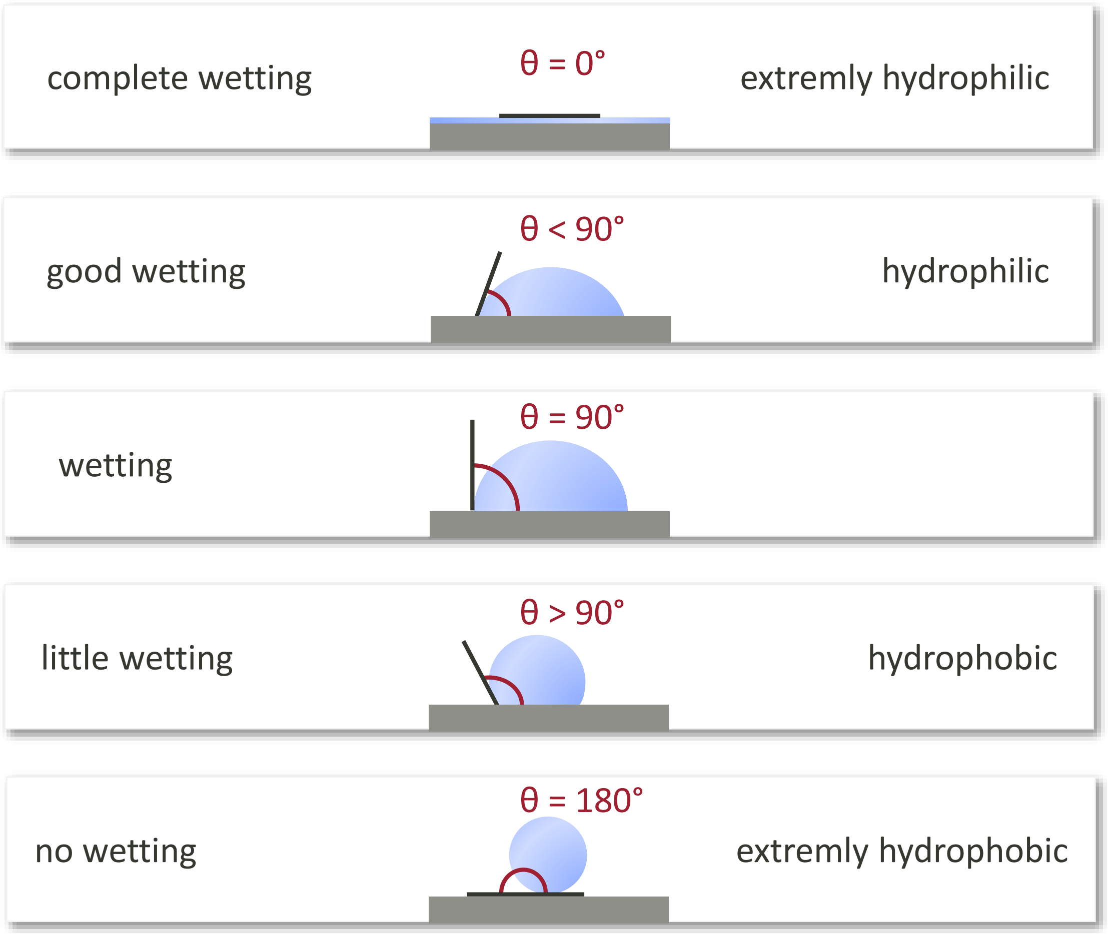
SVT Spinning-Drop-Tensiometer

HGC Humidity Generator and Controller

Challenge:
Measuring on Structured Test Areas

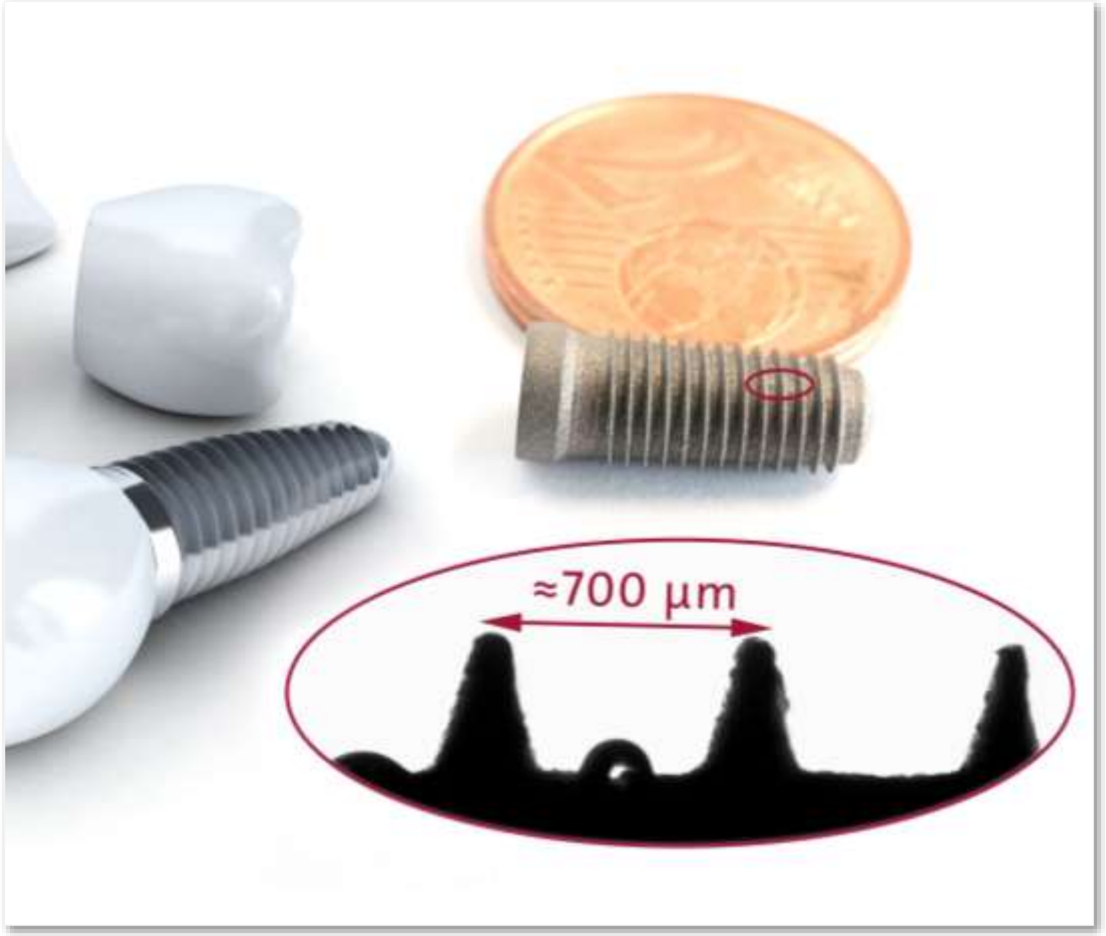
Surface Measurement: Contact Angle

- the contact angle θ is measured at the contact line between
  - a solid substrate
  - a liquid drop and
  - a surrounding gaseous phase
- the static contact angle gives an indication about the wetting and absorption of this substrate-liquid-combination
- contact angles can be used to classify solid substrates according to their wetting behaviour against water
- contact angle measurements with multiple known test liquids allow to calculate
  - surface energy of the substrate incl. polar and dispersive parts



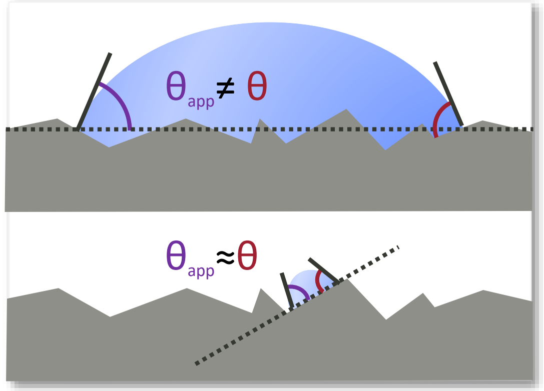
Challenge: Structured Surface

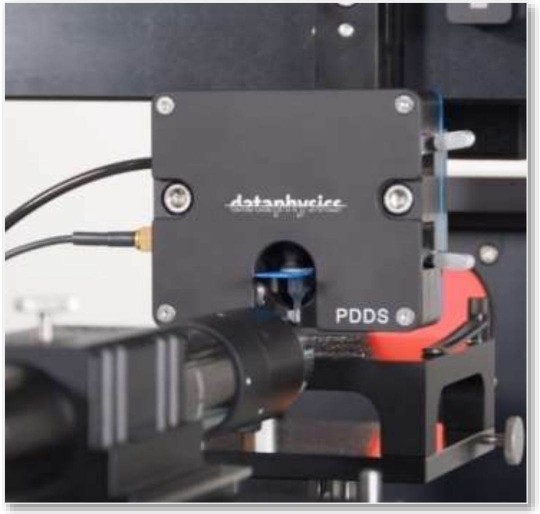
- → many real-life products have a structured surface
- → for example, dental implant screws only allow tests in an area of about 700 µm
- → for wetting experiments, that means extremely small drops are needed for contact angle measurements



Solution: Picolitre Drops

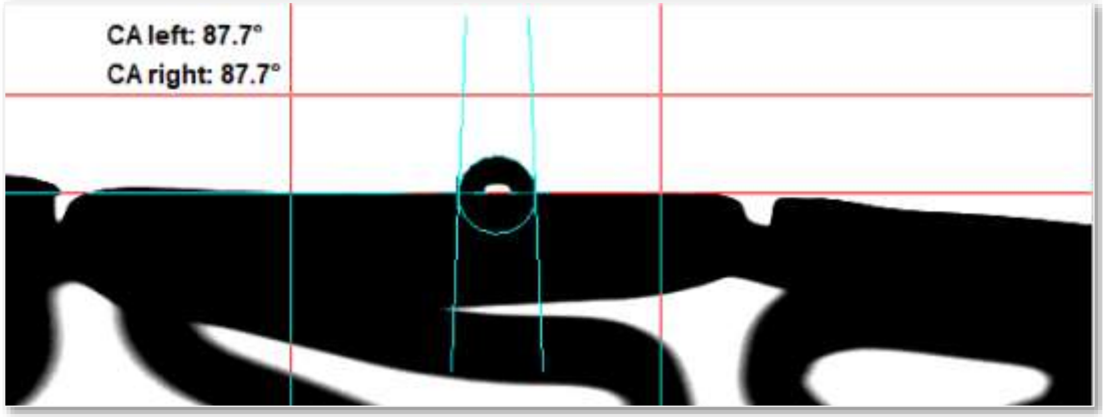
- when big drops are dosed onto structured surfaces, the measured contact angle θ_{app} does not correspond with the actual contact angle θ
  - measurement is not valid
- solution: dosing small drops corresponding to the structured surface
  - the measured contact angle θ_{app} is similar to the actual contact angle θ
  - valid measurement
- the PDDS picolitre dosing system together with an OCA contact angle meter can realise small drop sizes in the picolitre range

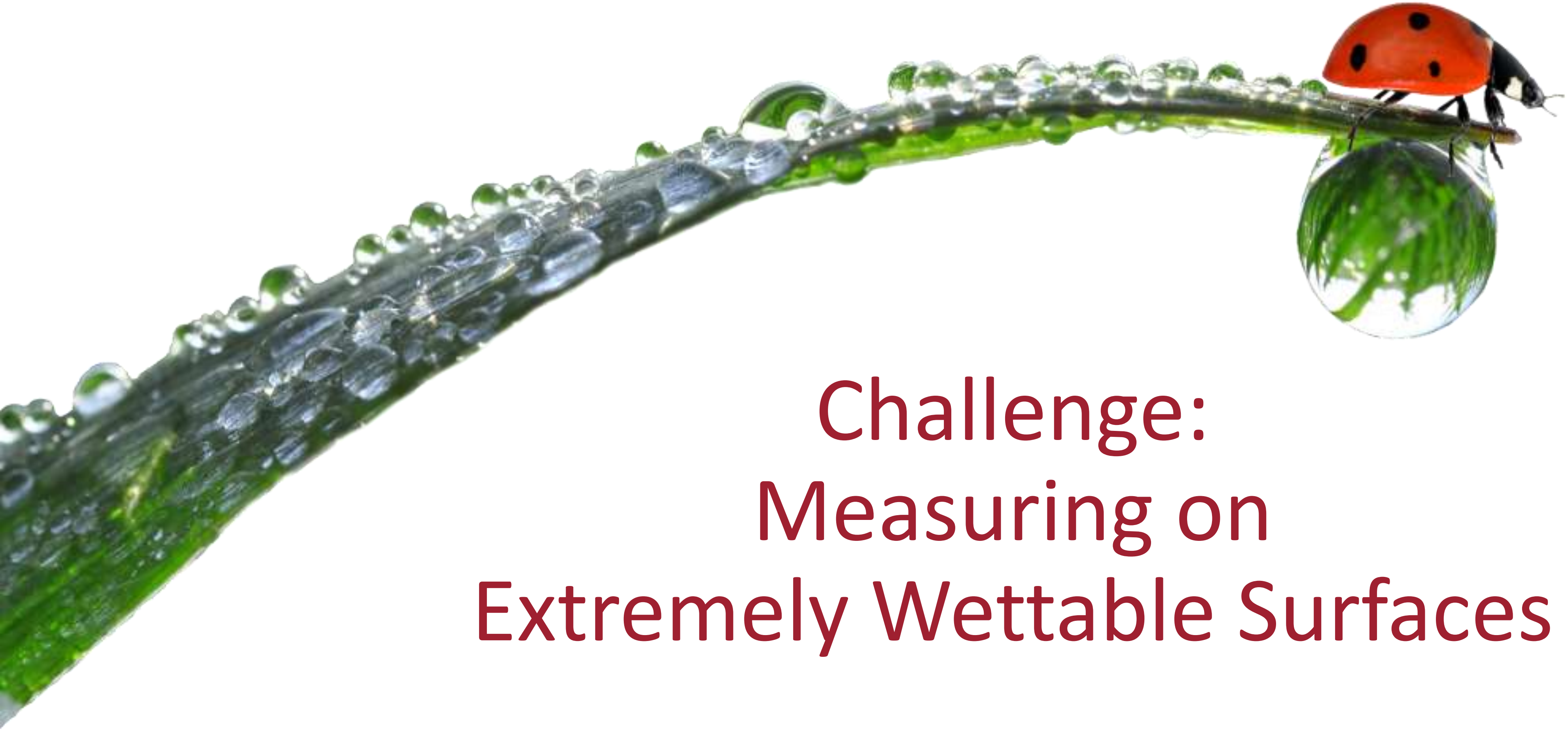




Solution: Picolitre Drops

- An OCA contact angle meter from Dataphysics Instruments can be fitted with a PDDS picolitre dosing system
  - uses disposable cartridges
    - no time-consuming cleaning required
    - no cross-contamination
  - acoustic pulses generate smallest droplets
    - minimal dosing volume: 30 to 380 pl, depending on the liquid's viscosity
  - dispensing frequency up to 1000 Hz
  - automated wetting analysis
  - allows emulation of real production processes, e.g., printing

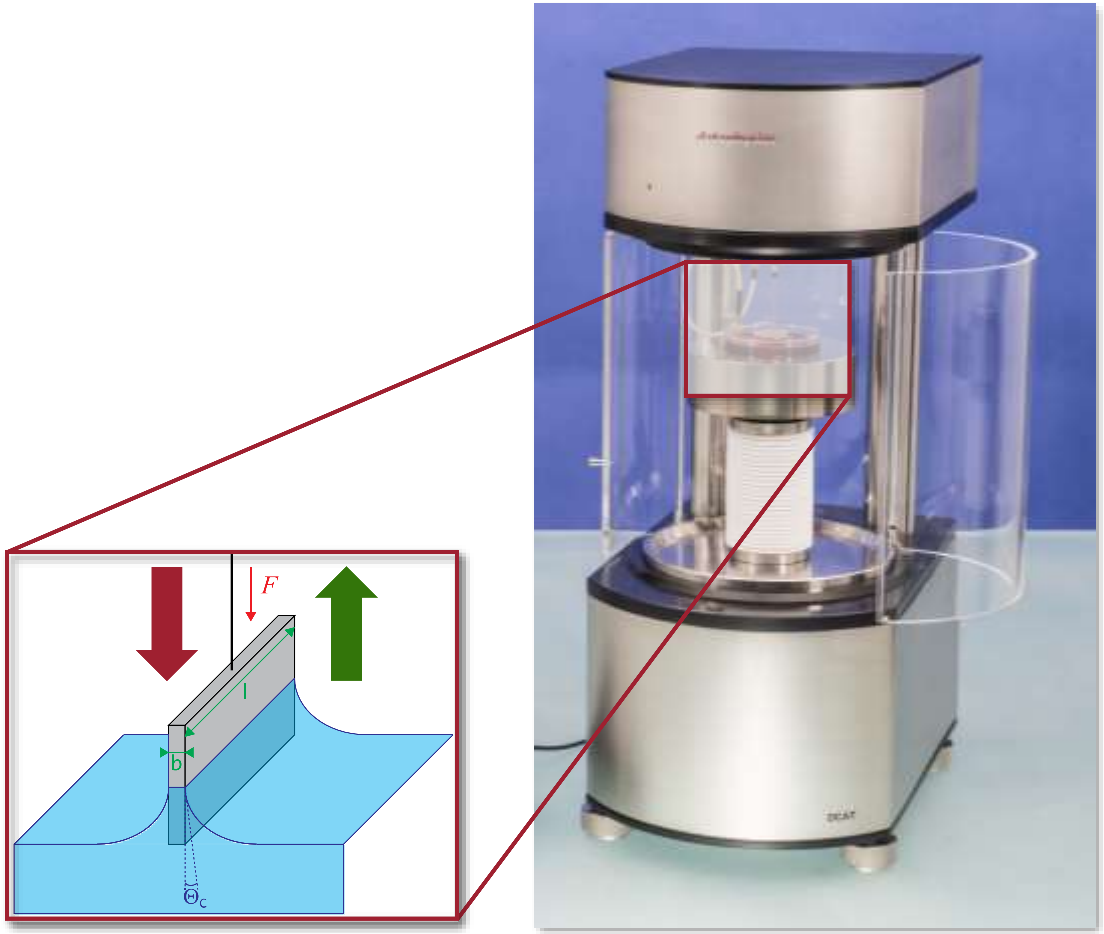




Challenge:
Measuring on
Extremely Wettable Surfaces

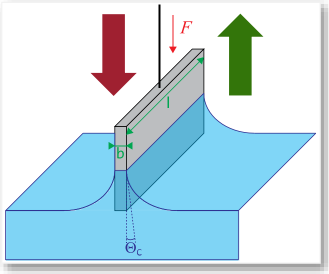
Surface Measurement: Tensiometry

- DCAT Tensiometers from DataPhysics Instruments can measure dynamic contact angles, i.e., contact angles in movement
- do to so, the solid substrate is suspended from a precise balance, located at the top of the device and dipped into and pulled out of the liquid
  - when dipped into the liquid: advancing contact angle θ_{adv}
  - when pulled out of the liquid: receding contact angle θ_{rec}



Surface Measurement: Tensiometry

- experimental set-up:
  - use test liquid with known surface tension σ
  - suspend solid sample from the device's balance
  - tare device balance after solid sample was attached to the balance and before it is dipped into the liquid
  - to be determined: dynamic contact angles θ of the solid sample body
- force F at the balance at an extrapolated height = 0 can also be described as $m_{h=0} \cdot g$
- width b and length l of the sample body yield the wetted length L (= 2l +2b)



- Wilhelmy equation:

$$\cos \theta = \frac{m_{h=0} \cdot g}{L \cdot \sigma}$$

- θ = advancing/receding contact angle
- $m_{h=0} \cdot g = F$ = mass at height 0
- h = height above the surface
- g = gravitational constant
- L = wetted length of the solid sample body
- σ = known surface tension of the liquid

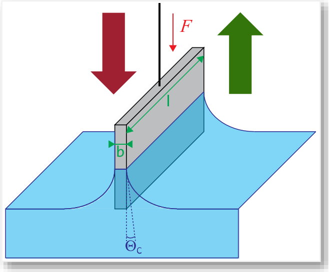
Surface Measurement: Tensiometry

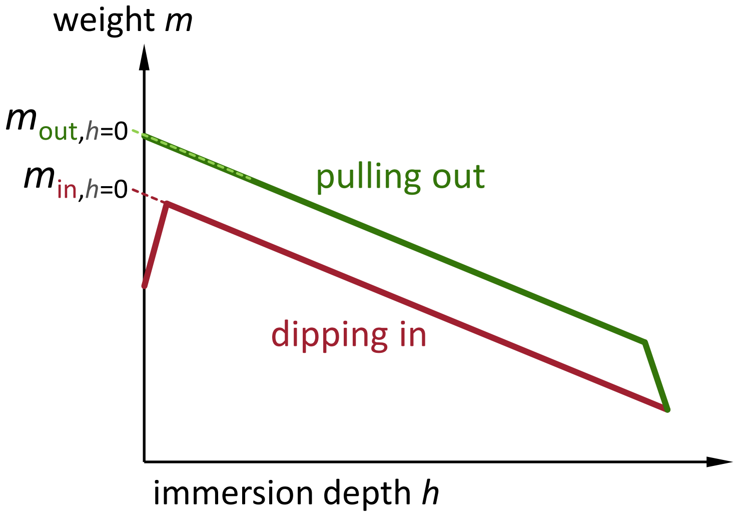
→ the software records the weight change that occurs while the sample is dipped into and pulled out of the test liquid

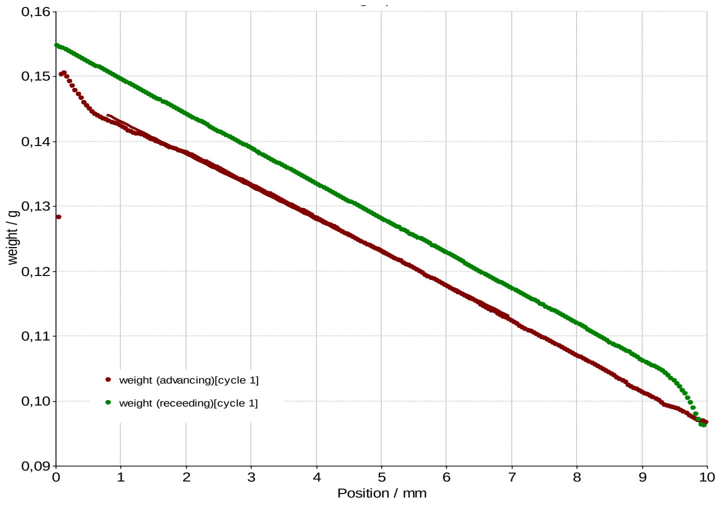
→ it then calculates

→ the advancing contact angle θ_{adv}

→ and the receding contact angle θ_{rec}







Challenge: Hydrophilic Surfaces

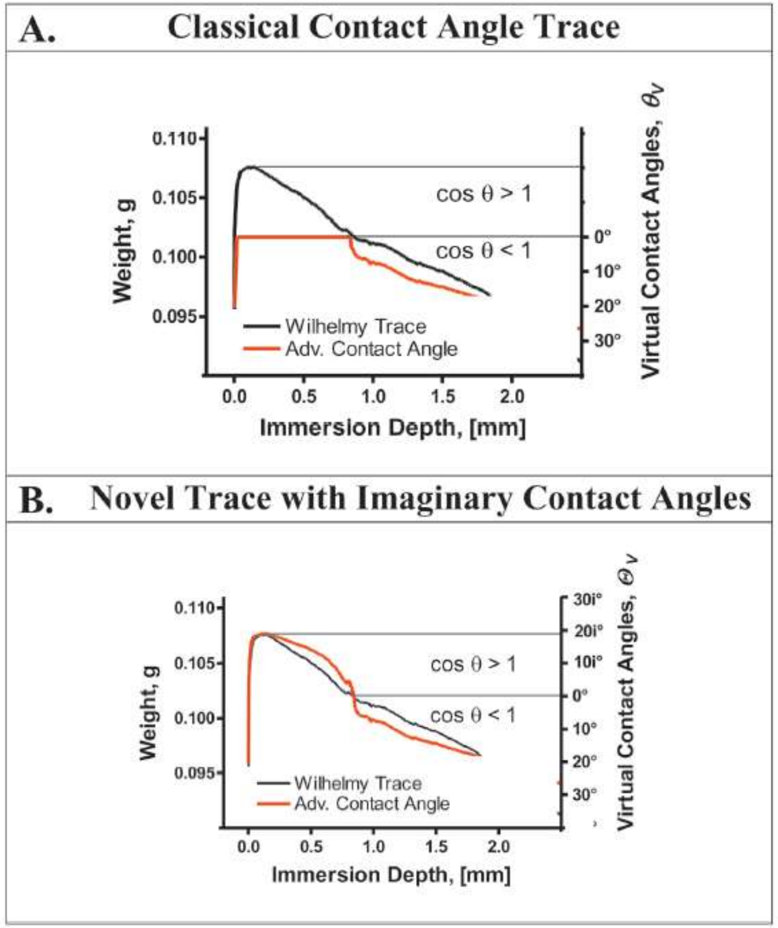
$$\cos\theta = \frac{m_{h=0} \cdot g}{L \cdot \sigma}$$

- If surfaces are very wettable using water, they are called extremely hydrophilic
- such behaviour is desirable in many applications, e.g., when developing dental implant screws
  - this can be observed as the liquid spreads quickly on the surface
- measurement challenge:
  - when measuring extremely hydrophilic surfaces, experiments often yield values greater than 1 for cos θ
  - the Wilhelmy equation cannot be solved, as cos > 1 = invalid
- question: how to quantitatively analyse hydrophilic samples?

Solution: Imaginary Contact Angle

$$\cos \theta i = \frac{m_{h=0} \cdot g}{L \cdot \sigma}$$

- in experiments, the values for cos θ can be larger than 1; this happens for many extremely hydrophilic surfaces, showing complete wetting
- no real number can yield a result for cos greater than 1
- solution: use a complex number to solve the Wilhelmy equation and calculate the 'imaginary' contact angle
  - a complex number is defined as
    $z = a + bi$, where
    - a = real part
    - b = imaginary part
    - i = imaginary unit, solution of the equation $i^2 = -1$
  - using cos (θ*i*) instead of cos (θ), the Wilhelmy equation is solvable even for values above 1



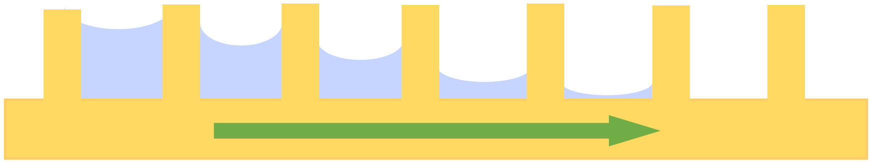
The concept of imaginary contact angles has been introduced by H. P. Jennissen (Materialwiss. Werkstofftech. Mater Sci Eng Technol. 2011; 42: 1111–7)

Solution: Imaginary Contact Angle

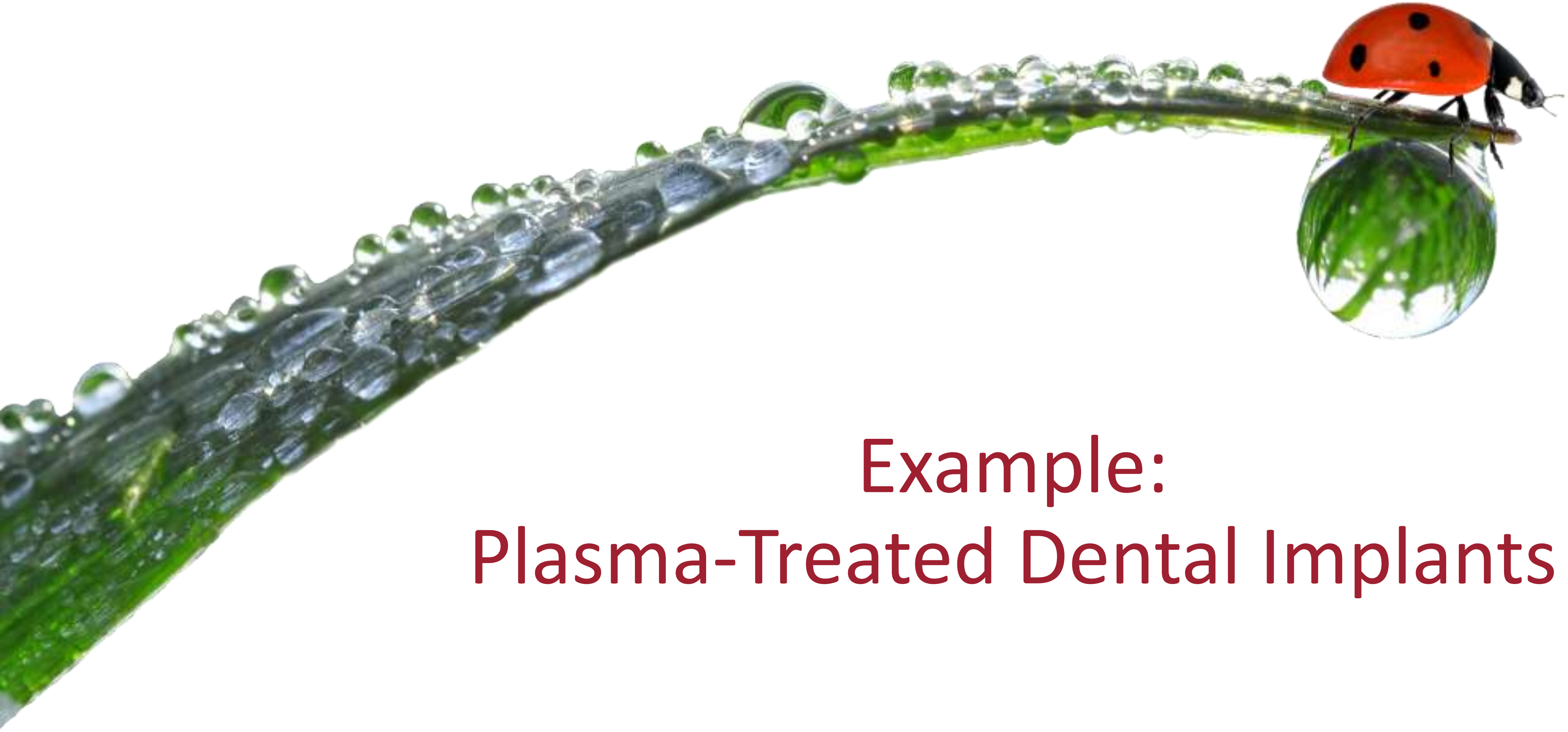
$$\cos \theta_i = \frac{m_{h=0} \cdot g}{L \cdot \sigma}$$

- why are some surfaces very wettable?
- rough or structured surfaces, in particular, experience additional wetting caused by, e.g., capillary forces
- using imaginary contact angles, these surfaces can be characterised and compared

A) Filling-up of rough surface



B) Complete wetting



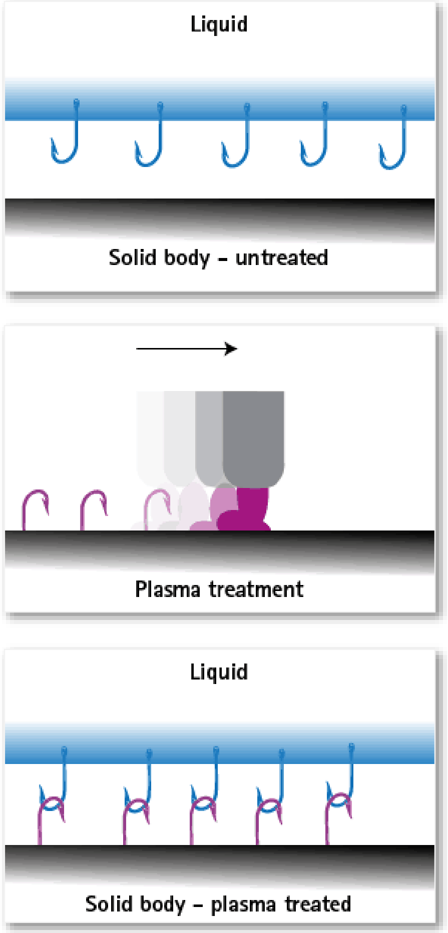
Example:
Plasma-Treated Dental Implants

Example: Dental Implants

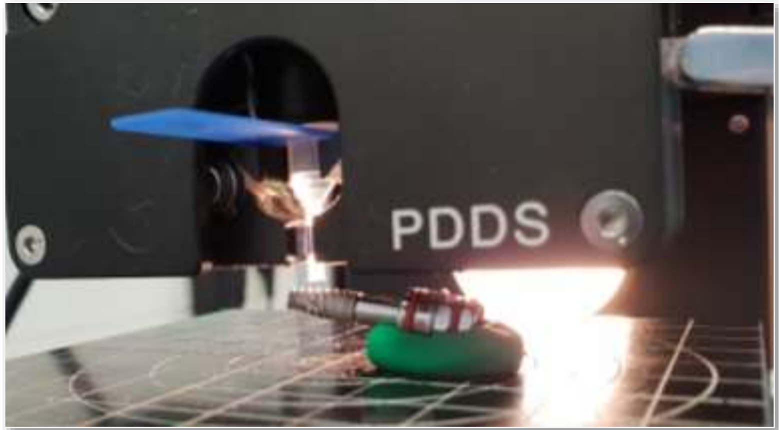
- challenge: implant surfaces need to be accepted by body tissue
- in experimental measurements, this translates as a surface with …
  - … low contact angle
    - complete wetting desired
  - … high surface energy
    - parameter to verify a successful pre-treatment and cleaning of the implant
    - high surface energy leads to better contact between body tissue and implant
    - estimation of the wetting behaviour and adhesive properties

Example: Plasma-Treated Dental Implants

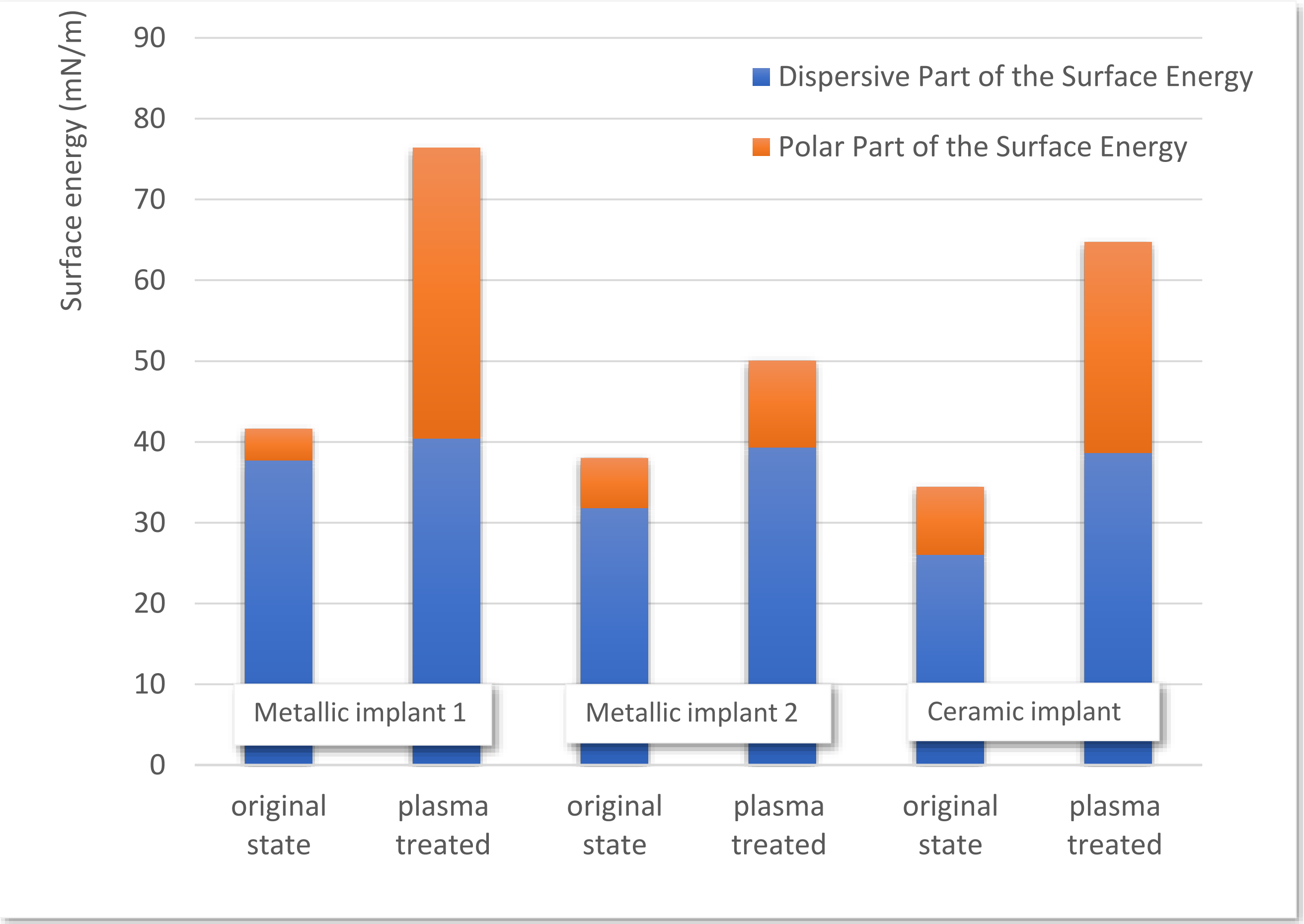
- problem: most polymers, and many other surfaces, are not wettable enough for further processing
- solution: pre-treating surfaces with a low surface energy and high contact angles (also called “functionalisation” or “activation”)
  - better wettability (higher contact angles)
  - raising polar part of the surface energy
  - ultra-fine cleaning
  - optimisation of bonding processes
  - better acceptability of implants by body tissue



Example: Plasma-Treated Dental Implants

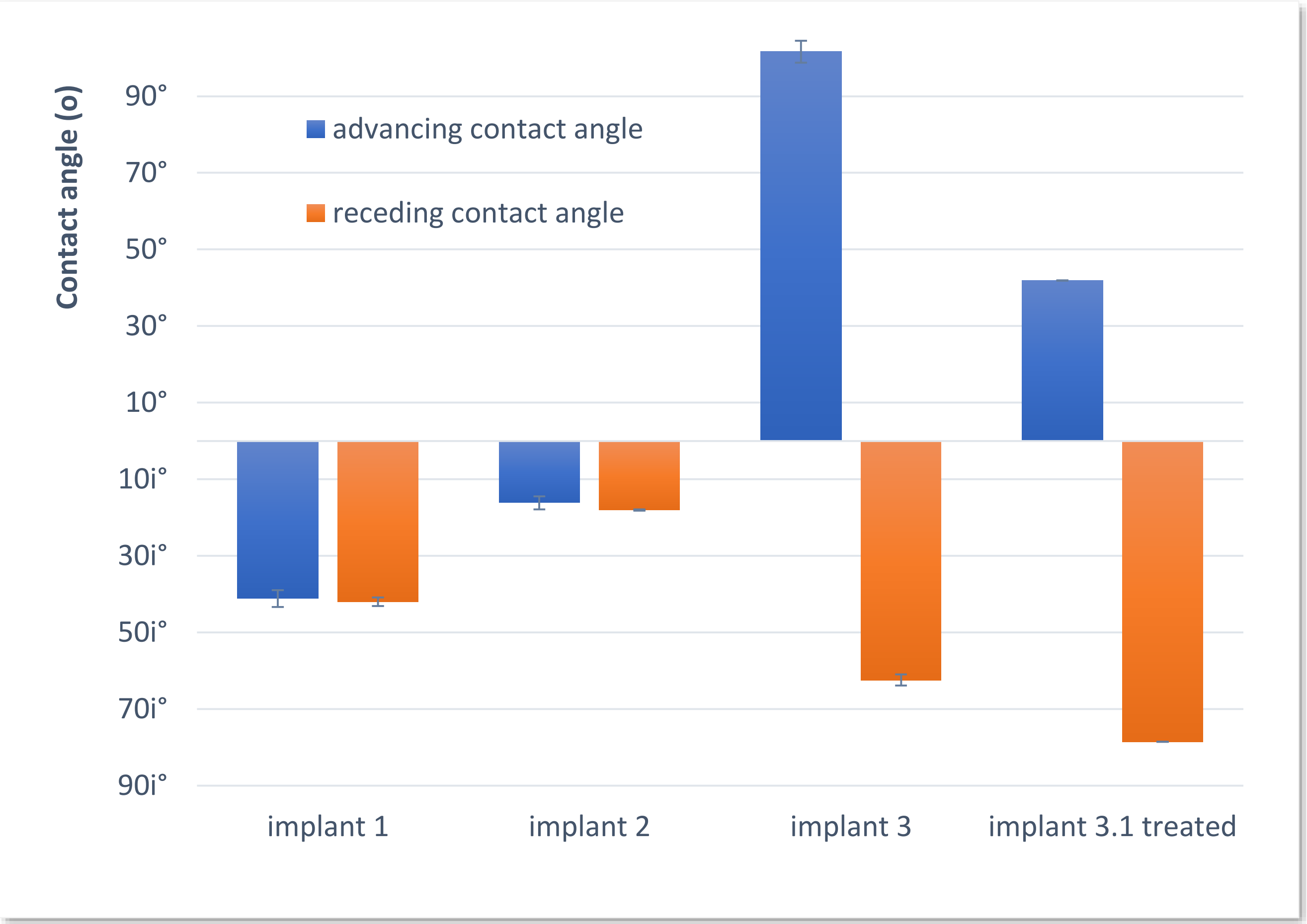


handheld plasma device piezobruch PZ3 from Relyon Plasma

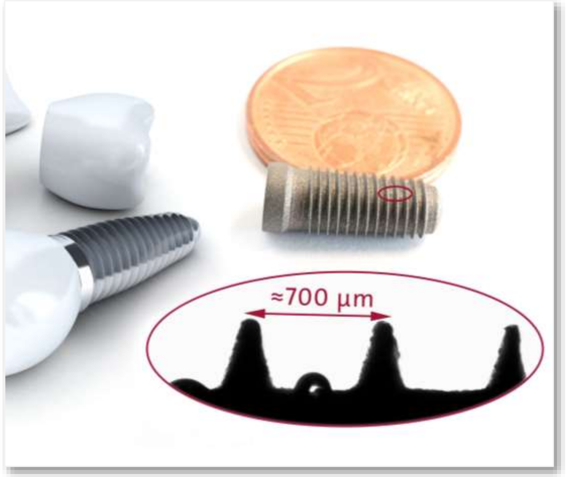


Example: Plasma-Treated Dental Implants

implant 3 and
implant 3.1 treated:
no spreading was
observed during
dipping in

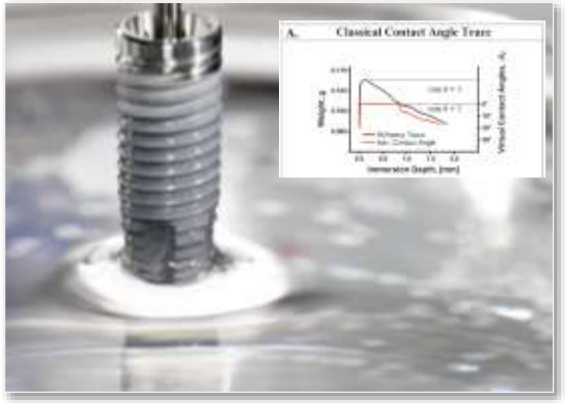


Summary: Plasma-Treated Dental Implants



Small Drop Dosing

- analyse structured substrate surfaces according to their wetting behaviour
- using static contact angle measurements



Imaginary Contact Angle

- quantitatively compare extremely hydrophilic substrates according to their wetting behaviour
- using dynamic contact angle measurements

Thank you!

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