New developments in print quality characterization

How topography analysis provides additional insights in combination with ink-substrate interaction studies

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dataphysics

Inkjet Academy 2020

DataPhysics Instruments GmbH

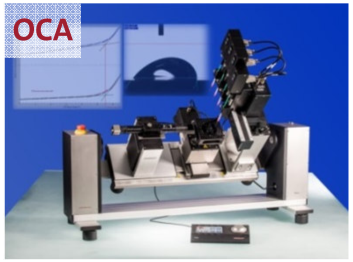
→ Manufacturer of high-quality measuring equipment for characterizing surfaces and interfaces...

dataphysics
Understanding Interfaces

→ Start-up in 1997:
Headquarters in Filderstadt with modern Application Lab & Training Center

→ Since 2018: US subsidiary in Charlotte, NC

→ Sales activities worldwide in more than 70 countries...



Optical Contact Angle measuring and contour analysis systems

Dyn. Contact Angle measuring instruments and Tensiometers

Spinning-drop Video-Tensiometers

Humidity Generator and Controller

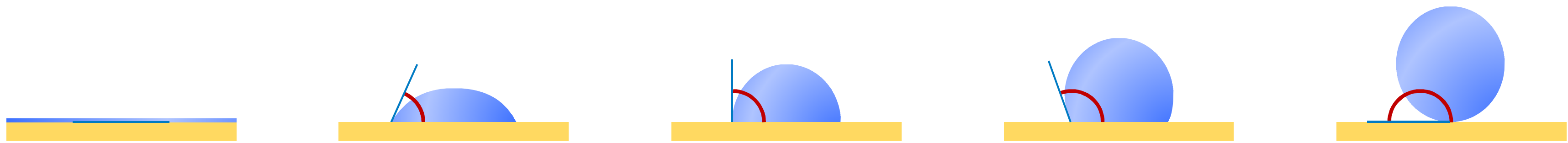
MultiScan stability analysis system

Surface Profile Analyzer

Print quality characterization for minimization of development costs

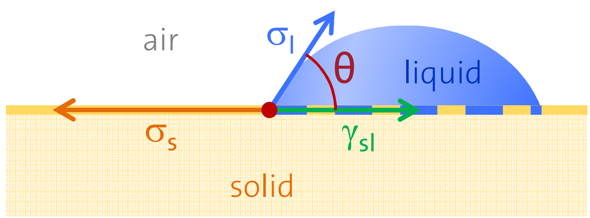
- Printing ink and substrate development are targeted towards an application-specific optimization of the ink-substrate interaction
- Study of ink-substrate interaction by **Wetting Analysis !!!**
  - **Measurement of contact angles**
  - Characterization of ink and substrate surface properties
    - Surface tension/surface energy
    - Prediction of wetting behaviour
  - Study of absorption behaviour
  - **Emulation of final printing process at early stage development**

Contact angle



$\theta = 0°$	$\theta < 90°$	$\theta = 90°$	$\theta > 90°$	$\theta = 180°$
complete wetting (spreading)	good wetting	wetting	bad wetting	no wetting

→ The contact angle θ is determined by the surface tension of the liquid σ_l & the surface energy of the solid σ_s ...



→ for the static contact angle θ (motionless front of the liquid) it holds:

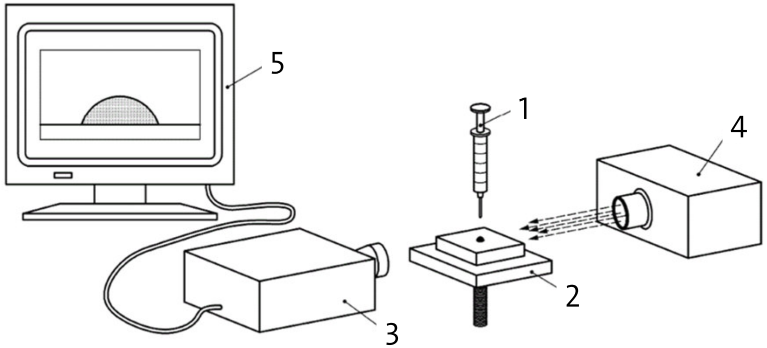
$$\sigma_s = \gamma_{sl} + \sigma_l \cdot \cos\theta$$ **Young equation**

→ describes the equilibrium of forces at the 3-phase contact line

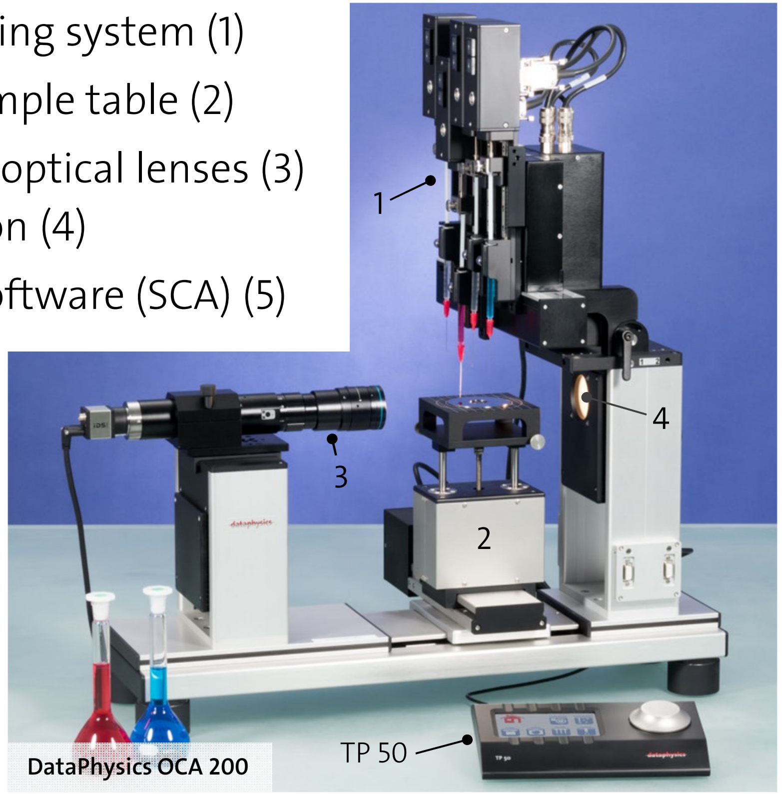
→ γ_{sl} is the solid-liquid interfacial energy, which depends on the “compatibility” of the bonds of the solid and those of the liquid...

Contact angle measurement with optical contour analysis system

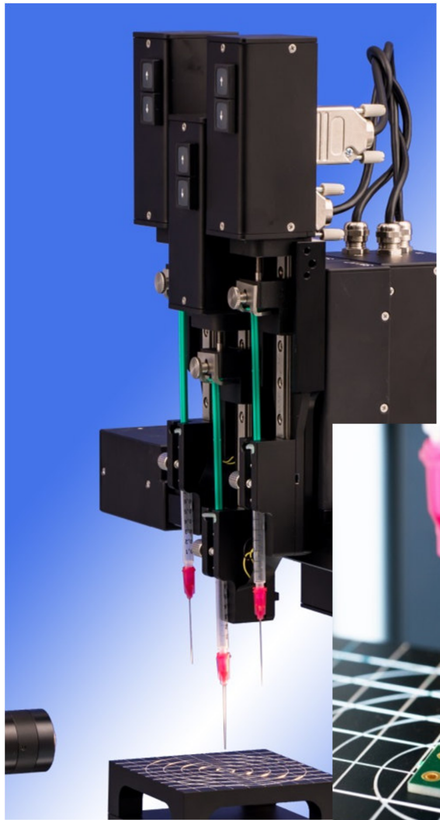
- Defined dosing of liquid drops — by suitable dosing system (1)
- Positioning of solid substrates — by movable sample table (2)
- Optics for capturing drop images — by camera and optical lenses (3) and illumination (4)
- Software for system control and image processing — by according software (SCA) (5)

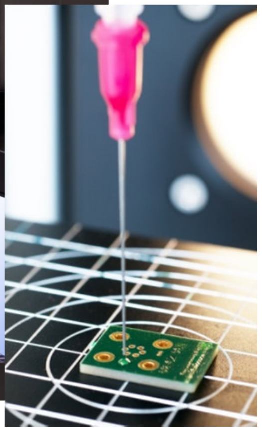


Schematic Figure from DIN 5560

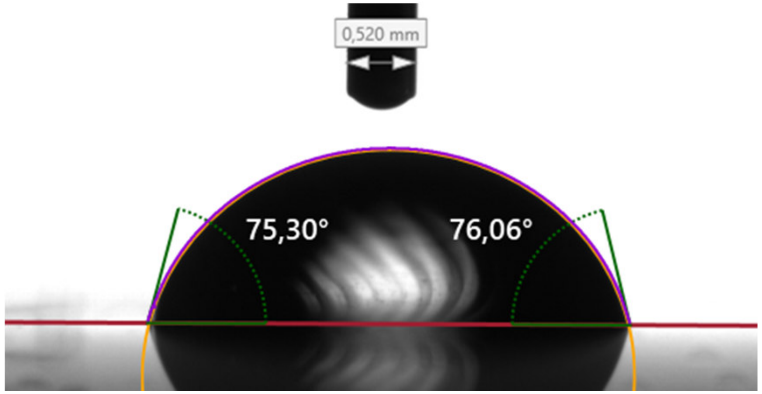


Contact angle measurement with optical contour analysis system



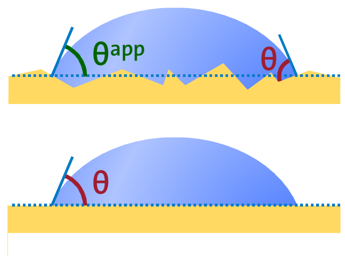


- → Sessile drop is dosed on substrate, conventionally by syringe in electronic syringe module
- → When contact area is constant:
  - → Automatic substrate line & drop contour detection, contour fit with appropriate model (e.g. ellipse) & direct contact angle calculation



Contact angle measurement on rough surfaces

- Conventional dosing of µl drops yields the 'apparent CA' θ^{app}
  - for rough surfaces: $\theta^{app} \neq \theta$ ('material CA')
  - for ideally flat surfaces: $\theta^{app} = \theta$



- **Drop size matters** for rough surfaces, like printing substrates!
  - ...independent of the dosing method!

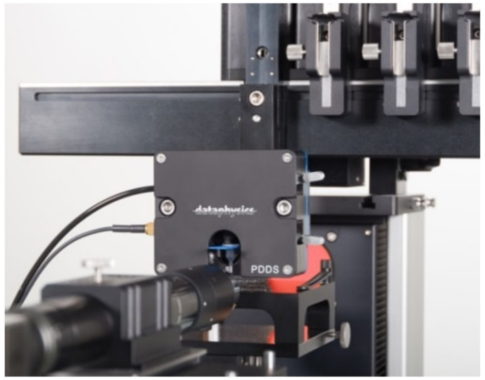
Water contact angles *on* *dosing system*	*drop size*	flat silicone wafer	paper (adsorbing)	coated paper (slightly adsobing)
conventional	*macroscopic*	69,0°	102,0°	55,9°
picodrop system PDDS	*macroscopic*	69,5°	104,0°	57,5°
	microscopic	68,5°	68,0°	38,7°

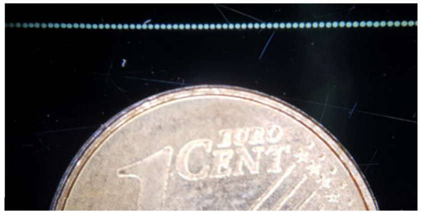
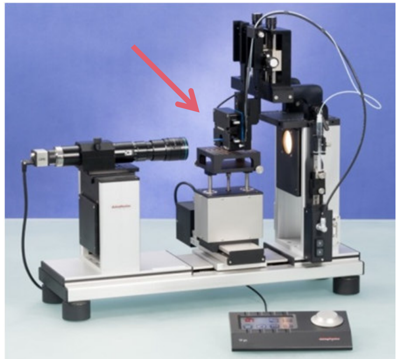
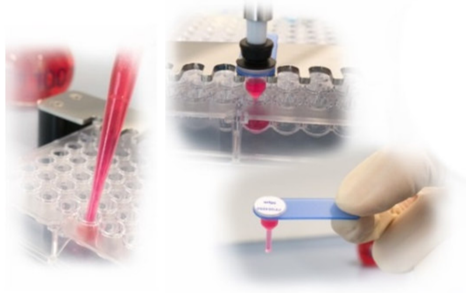
- **Characterize rough substrates with drop sizes as in the final application!!!**

On the Droplet Size and Application of Wettability Analysis for the Development of Ink and Printing Substrates, M. Grüßer, D. G. Waugh, J. Lawrence, N. Langer, D. Scholz, *Langmuir*, 2019, 35 (38), 12356-12365.

Picodrop Dosing System (PDDS) from DataPhysics Instruments

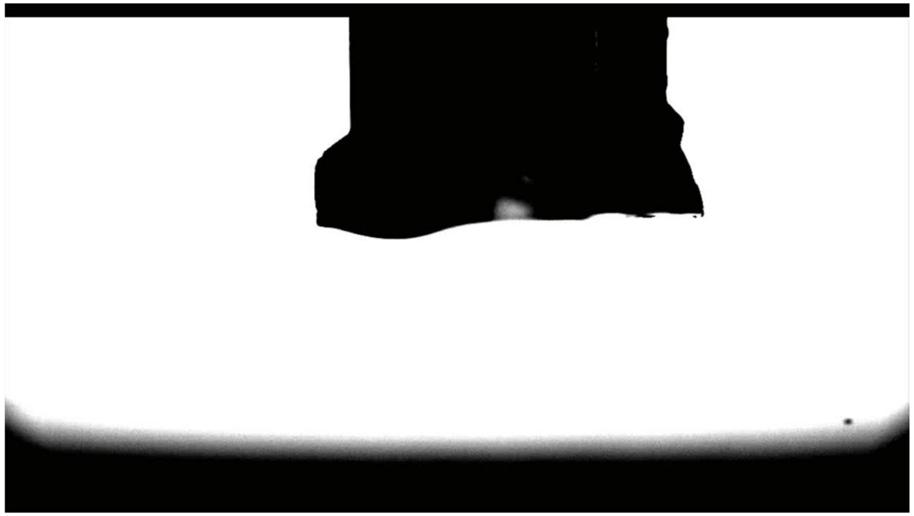
- dosing volumes down to 15...380 pl
  (depending on liquid viscosity)
- droplets dispensed via acoustic pulses from disposable cartridges
  - only small liquid amounts needed
  - no cleaning required
- dispensing frequencies up to 1000 Hz
- with electric table axes of OCA 200:
  - automated substrate movement
- **substrate characterization** for and **emulation** of real printing processes
  - drop size / dispensing frequency / substrate movement speed



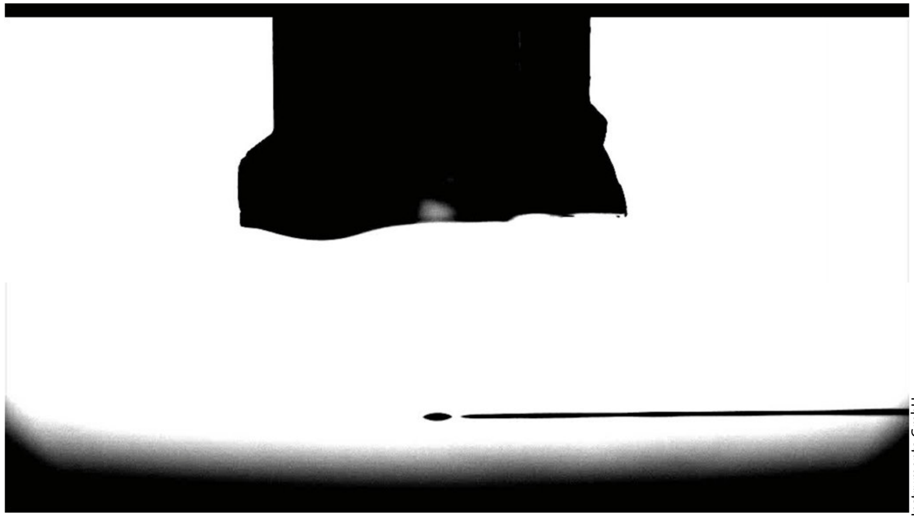





Application of the PDDS for printing studies

- → dispensing of individual drops
  - → allows wetting analysis



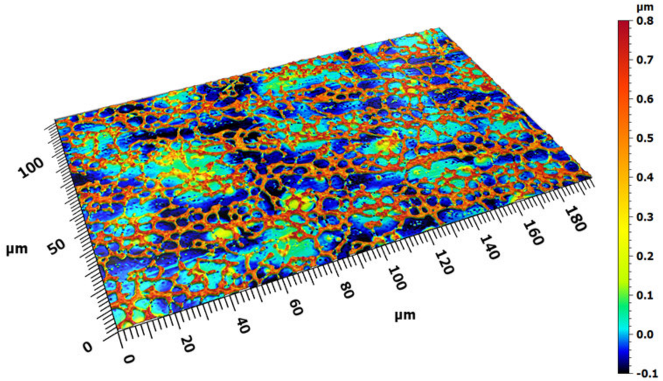
- → dispensing of connected drops
  - → emulates line printing



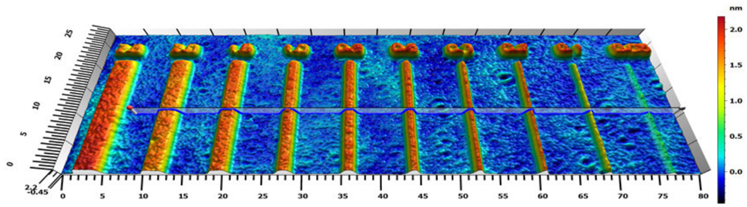
Additional insights trough topography analysis

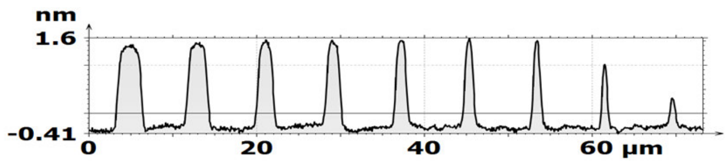
- Refinement of wetting analysis
  - roughness coefficient *r* of substrate for correction of the contact angle *according to* ***Wenzel***:

$$\cos \theta^{app} = r \cos \theta$$



- Evaluation of obtained print image
  - 3D representation & analysis…



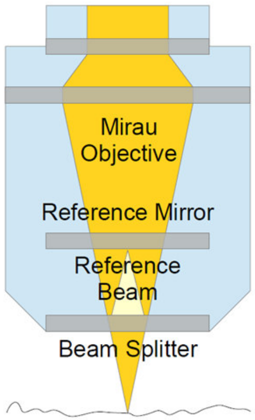


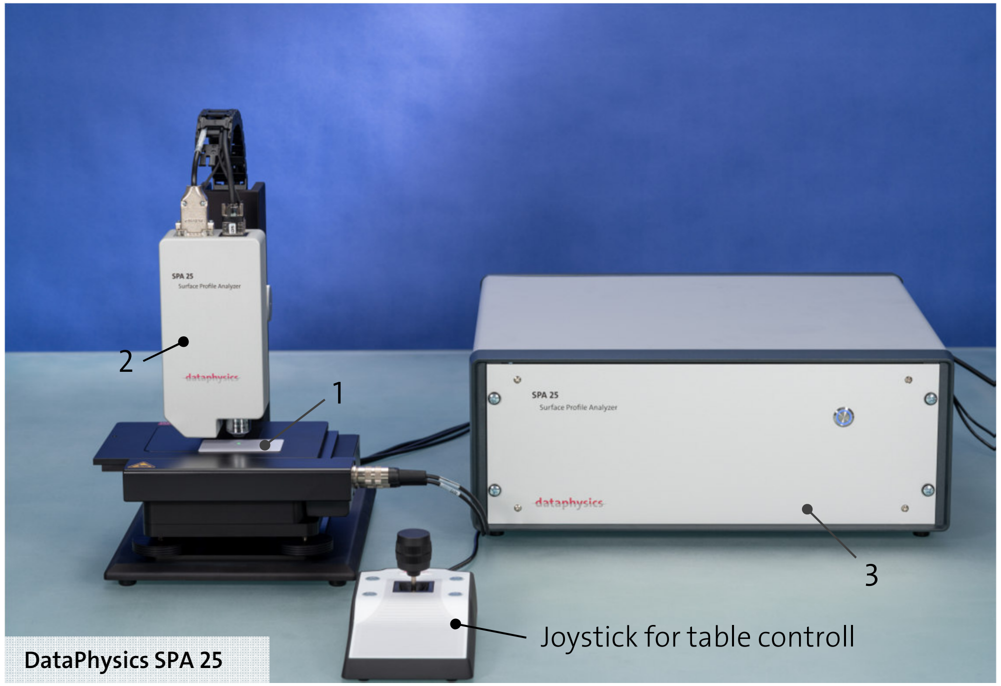
What happens after the ink has dried?

R. N. Wenzel, *Resistance of Solid Surfaces to Wetting by Water*. In: Ind. Eng. Chem. 28, 8, 1936, 988–994.

Surface Profile Analyzer (SPA 25) from DataPhysics Instruments

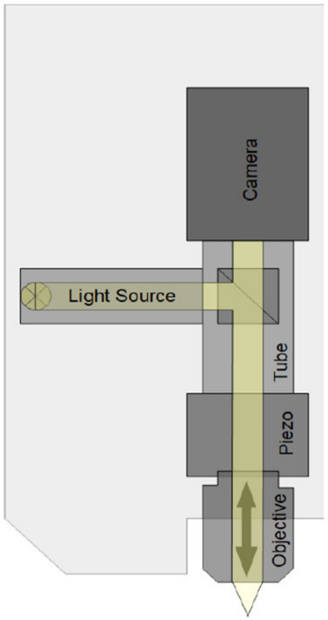
- Defined positioning of the sample (1) (automated table allows for stitching)
- Measurement head with optics (2)
- Powerful computer to for data processing (3)

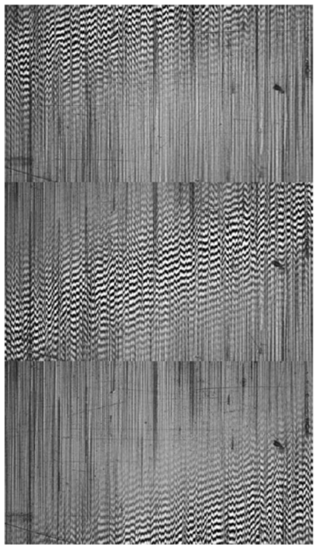


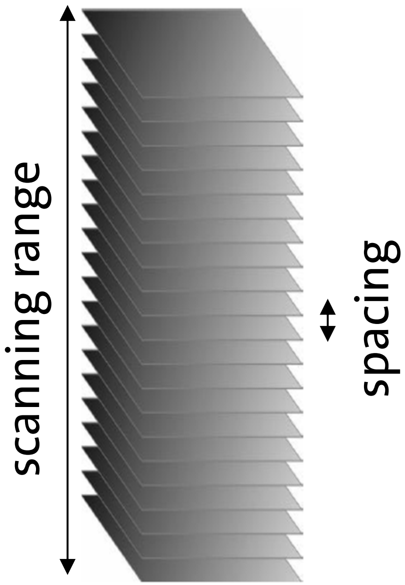


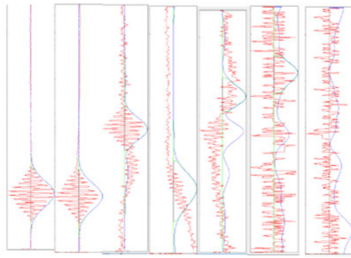
White Light Interferometer

Principle of White Light Interferometry







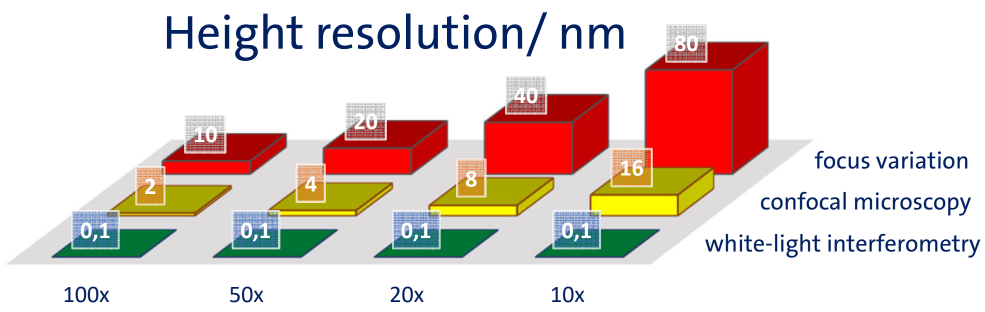


Spacing of 0.2 µm and smaller provides 0.1 nm hight resolution (EPSI Mode)

Up 500 GByte of image data will be processed with each scan

- The objective is moved in z-direction relativ to sample
- In the interference zone ther is a sinusoidal change of the light intensity
- This signal is analyzed -> determination of the 3d surface in high resolution

Resolution Comparison for Different Methods



magnification	2.5x	5x	10x	20x	50x	100x
camera	2 MP – 1900 x 1200 pixel / measuring points					
measuring field / mm²	7.3 x 4.6	3.7 x 2.3	1.8 x 1.2	0.91 x 0.58	0.37 x 0.23	0.18 x 0.12
point spacing / µm	3.8	1.9	0.96	0.48	0.19	0.1
WD / mm	10.3	9.3	7.4	4.7	3.4	2

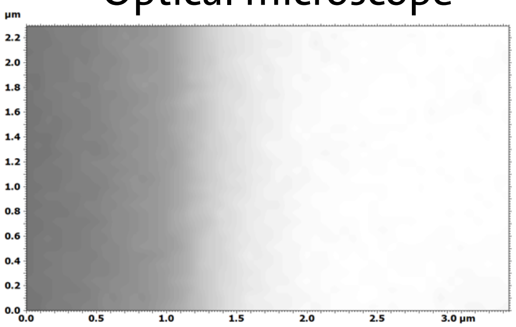
→ Resolution in Z-direction independent of objective

→ Highest resolution in Z-direction

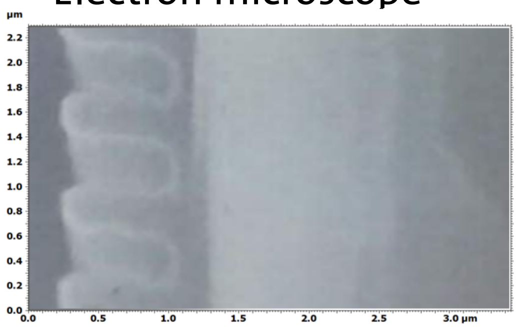
→ Resolution in X/Y-direction and field of view depends on objective

Different approaches to study the drops after the ink has dried

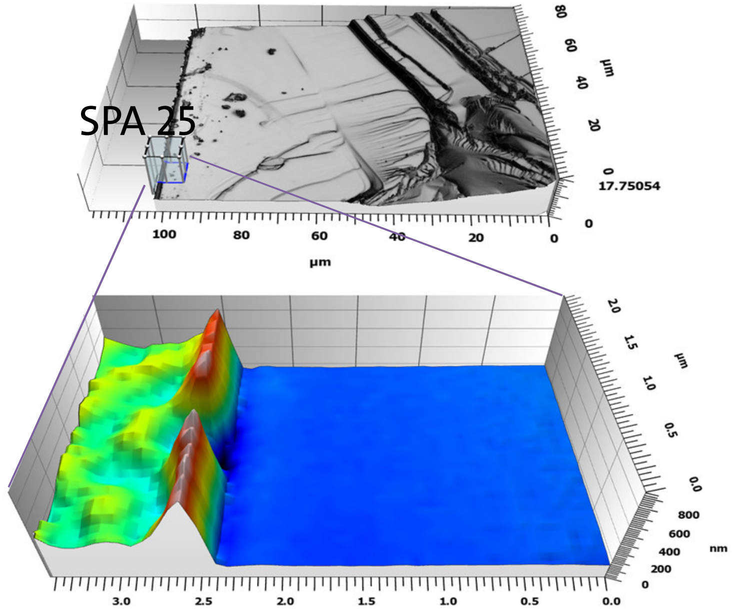
Optical microscope



Electron microscope



SPA 25

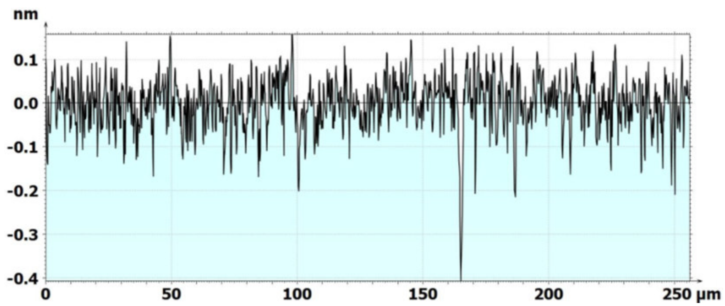


color coded 3D image/ partial area 3.5 x 2.3 µm²

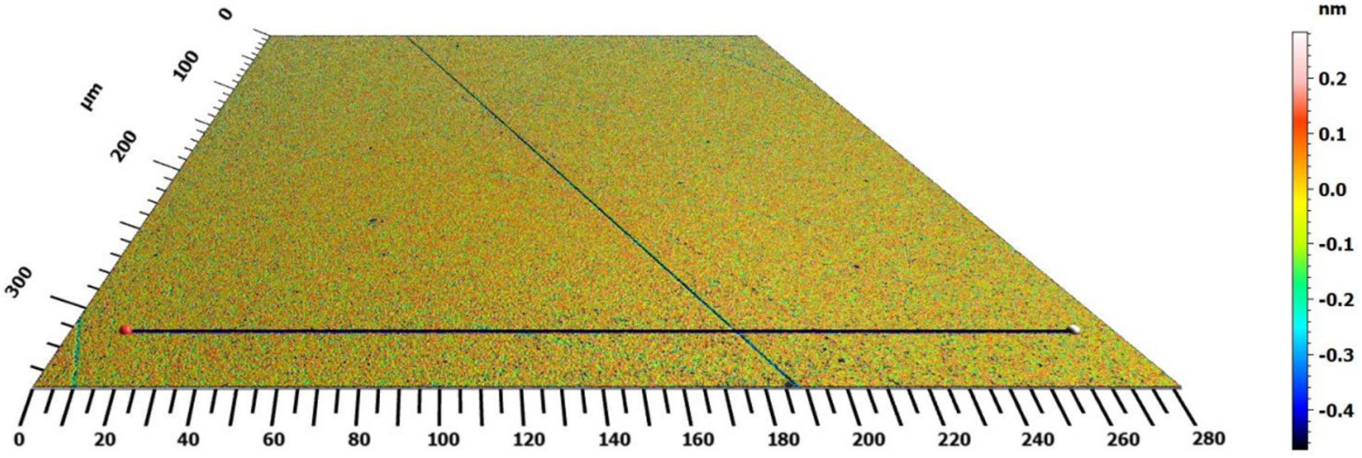
- Optical microscope: no details visible
- Electron microscope: high contrast on different materials in 2D
- SPA 25: fine details in 3D, no information about different materials

Example measurement on a polished waver

- 50x magnification
- Measured area 340 x 280 µm²
- Point density 0.14 µm
- Calculated from 10 measurements
- Surface structures with Rz smaller 1 nm

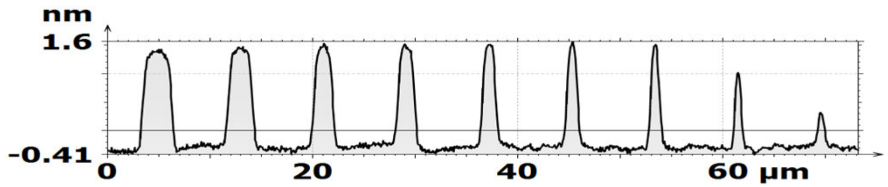


Geometry: Very smooth surface
Groove with 0.4 nm depths

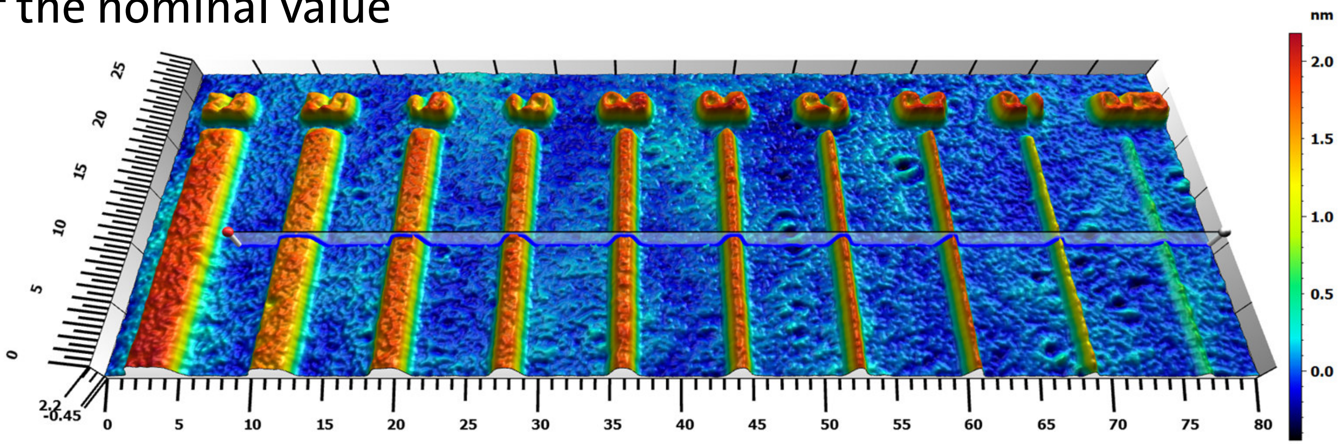


Example measurement on a standard with line structures

- 100x magnification
- Measured area 170 x 140 µm²
- Point density 0.07 µm
- On a 0.1 µm wide line structure the measured height value got down to app. 2/3 of the nominal value



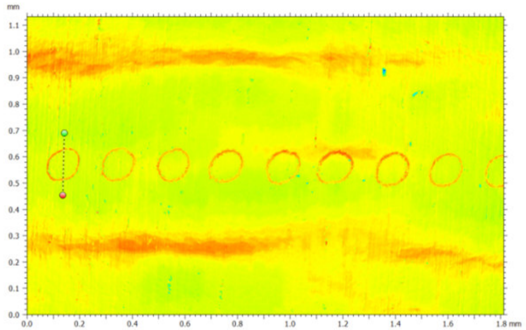
Geometry: Lines
1-2 nm height
1-5 µm width

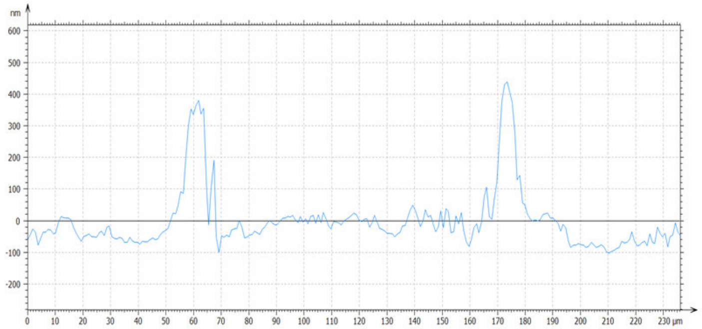


Drop Particle Distribution After Evaporation of the Liquid Medium

- → Directly after dosing spherical drops are observed

- → **Surface Profile analysis after the liquid medium evaporated!**
  - → All particles got washed to the border!





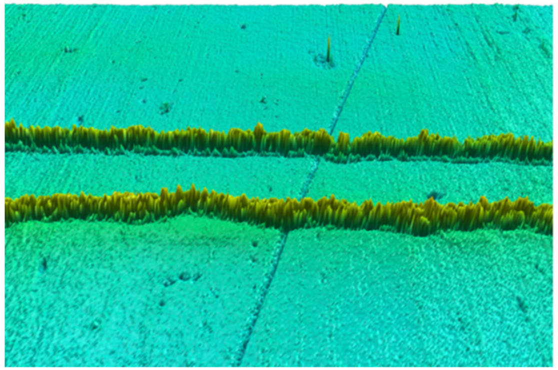
Geometry: Layer thickness 400 nm
Ring inner diameter 100 µm
Ring width 10 µm

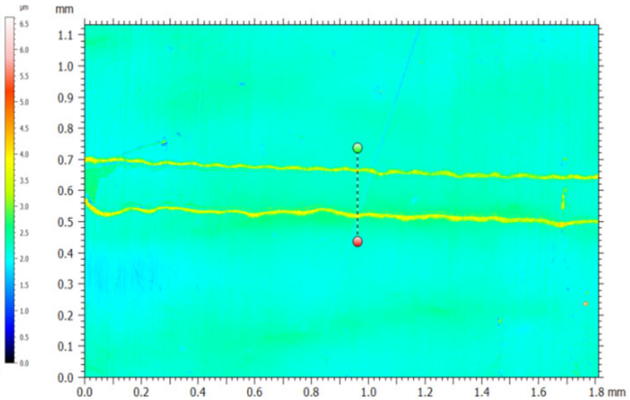
- → **A surface profile analysis can help to understand the drop geometry**

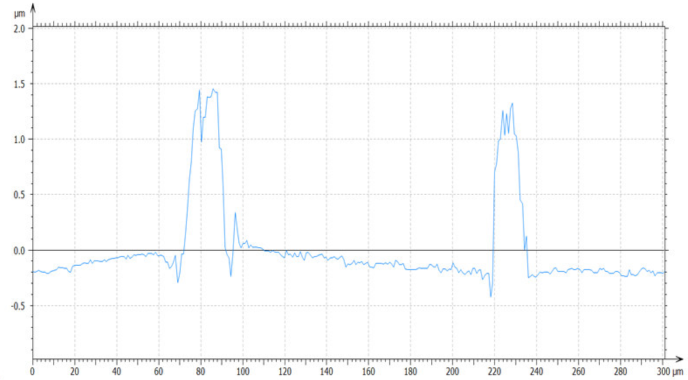
Drop Particle Distribution After Evaporation of the Liquid Medium

- → Directly after dosing a line is formed

- → **Surface Profile analysis after the liquid medium evaporated!**
  - → All particles got washed to the border and two particle lines are formed!





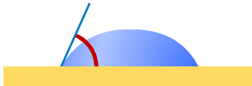


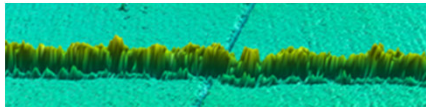
Geometry: Layer thickness 1.3 µm
Line width 15-20 µm
Line distance 130 µm

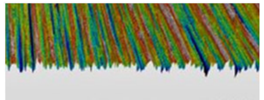
- → **A surface profile analysis can help to understand the line geometry**

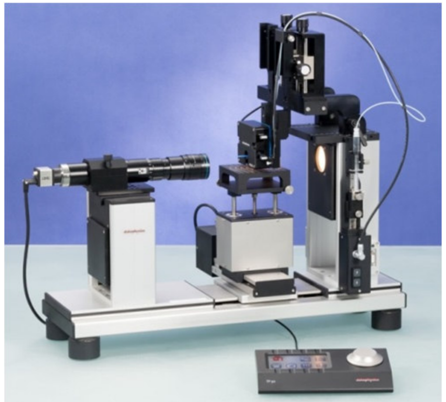
Understanding a printing process from dosing to the final print

- Wetting analysis
- Simulation of printing processes
- Measurement of diameters and geometry of printed wires
- Study of particle distribution for printed drops
- Estimation of layer thickness and homogeneity
- Roughness analysis

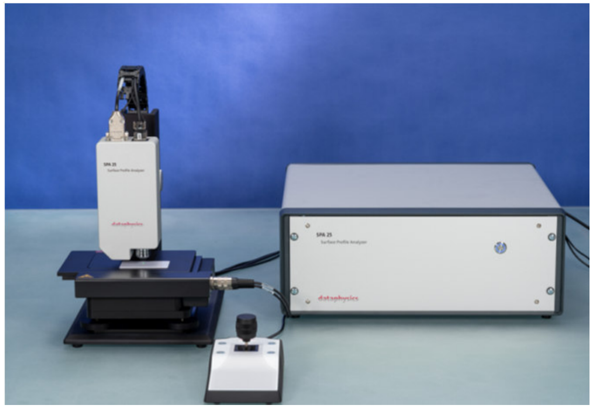




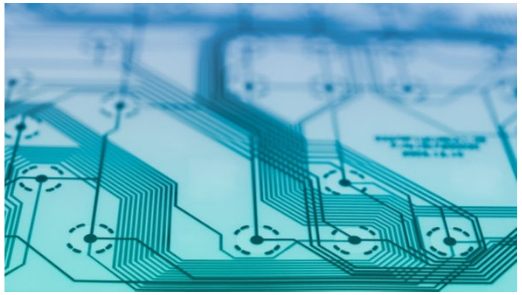




OCA 200

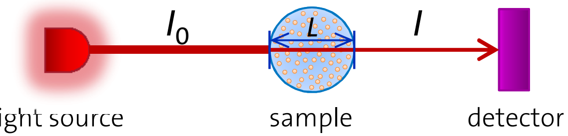


SPA 25



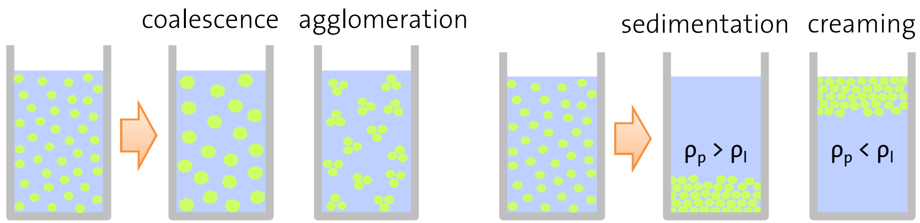
MultiScan Stability Analysis System (MS 20)

Optical stability analysis of liquid heterogeneous mixtures (suspensions, emulsions, foams)



Quantitative, objective results, non-intrusive, non-destructive method

Applications: Formulation optimization
Standard tests



Stability analysis and formulation optimization for inks

Thanks for your attention!

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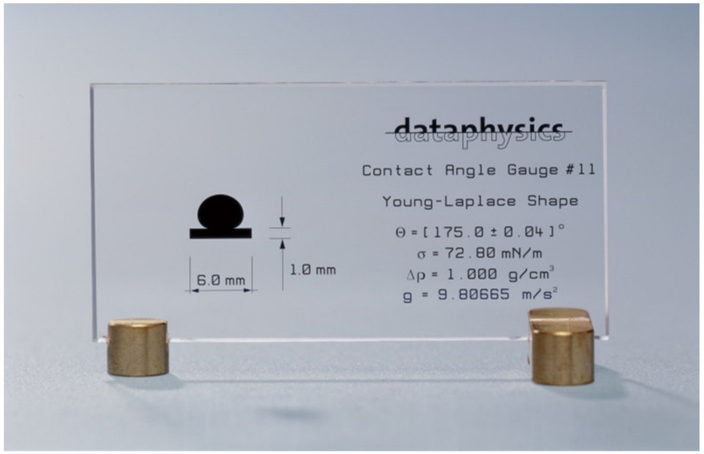

→ Contact angle standard (lithographic)

→ Surface profile analysis coloured

Summary: Print Quality Characterization

Ink-Substrate Interaction Study

Topography Analysis

- Contact angle measurement
- Simulation of ink jet printing

refinement

add. inform.

- Determination of roughness parameters
- 3D representation of the print image

- *Optical Contour Analysis System OCA 200 with Picodrop Dosing System PDDS*

- *Surface Profile Analyzer SPA 25*