



Because Surface Matters.™

## Hochauflösende Oberflächenanalytik an Polymer Substraten

SuSoS AG

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# Topic

- SuSoS
- Thin film and thin film analysis
- Polymers and polymer films: strengths and weaknesses
- Examples:
  - Detection of contaminants on surfaces
  - Activation of polymer surfaces
  - Roughness of a thin film by XPS
  - Chemical imaging and Orientation of Thinfilms
- Summary



✓ SuSoS because surface matters.  
A lot.

- We focus on those 50 nanometers that make the difference, so our customers can take care of everything else.



✓ SuSoS because surface is tough.

- But solutions must be simple.



✓ SuSoS because truly  
understanding is most of the  
solution.

- Guiding customers through hard decisions is key to long-lasting relationships.

## SuSoS Functional Polymer Thinfilms

### – Applications

- Medical Devices
- Microfluidics
- Diagnostics
- And many more...

### – Functionalities

- Non-Fouling
- High Lubricity in wet and dry state
- Adhesion of anything to anything
- Selective bonding
- And even more...

# Thin film and thin film analysis

- At SuSoS we focus on the **topmost atomic/molecular layers** of a product
- Surface Analysis Techniques at SuSoS:
  - X-ray Photoelectron Spectroscopy → **qualitative and quantitative** evaluation of chemical/atomic composition
  - Time of Flight Secondary Ion Mass Spectrometry → qualitative chemical/molecular composition
  - Variable Angle Spectro Ellipsometry → Thin Film Thickness
  - Micro Tribology and Indentation → Friction and Lubricity
  - Microscopy/AFM → Optical Appearance and Surface Roughness
  - Particle Counting → Amount of Particles in Solutions

Method	X-Ray Photoelectron Spectroscopy	Time of Flight Secondary Ion Mass Spectrometry	Energy Dispersive X-Ray Spectroscopy
	XPS	ToF-SIMS	EDX
Information depth	5-10 <b>nm</b>	1-2 <b>nm</b>	1-3 <b>µm</b> (= 1'000-3'000 <b>nm</b> )



# Polymers and polymer films: strengths and weaknesses

## Why Polymers?

- As Substrate
  - Often used in Medical Devices, Diagnostics or Microfluidics
  - Injection molded substrates are flexible, easy to produce with high accuracy and very small features and structures
  - High throughput with constant quality
- As Thin Film Coating
  - Functional groups can be easily exchanged and tuned to customer needs
  - With the right binding group polymer thin films can be bound to any substrate, even polymers
  - High flexibility, throughput and tunability

## What obstacles in analysis can occur?

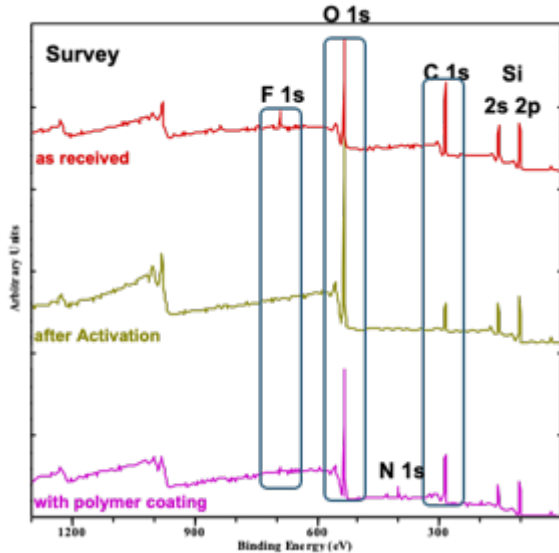
- Lack of Conductivity
- Carbon is Carbon
- Roughness
- De-gassing behavior

## How can some of these be overcome for example with XPS?

- charge compensation
- different binding partners = different binding energy
- Nano morphology can be analyzed

# XPS Standard measurements

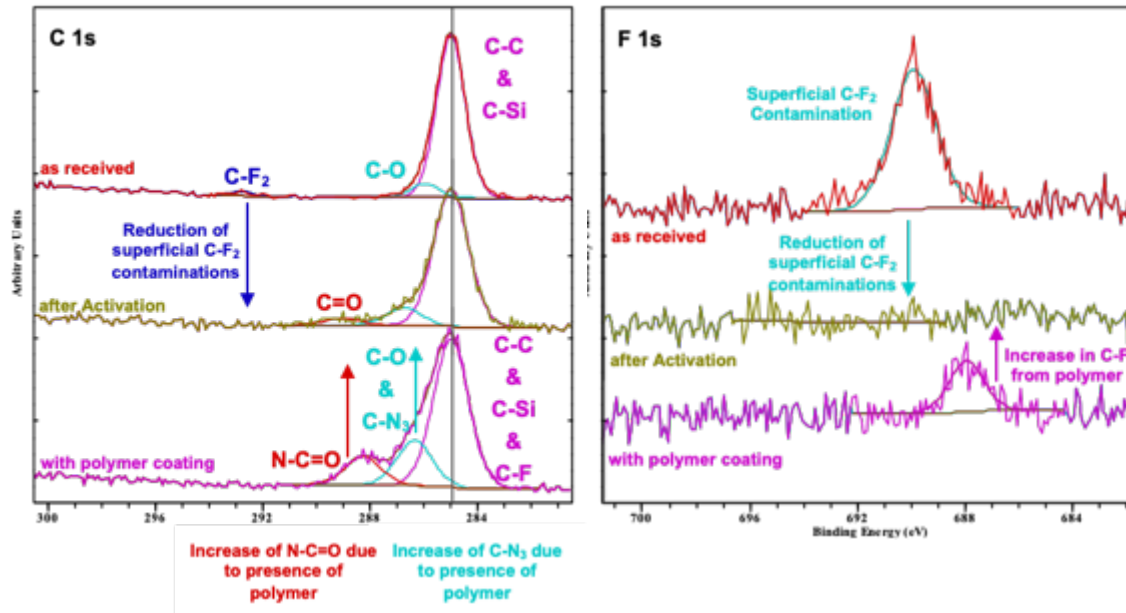
- Detection of contaminants on surfaces → Silicone Substrate with superficial C-F<sub>2</sub> Contamination
- Activation of polymer surfaces → O<sub>2</sub> plasma treatment reduces contamination levels and activates surface for further treatment/coating
- Evaluation of Polymer Coatings on Polymer Substrates → Non-fouling coating on silicone substrate can be detected due to additional coating related elements and binding groups



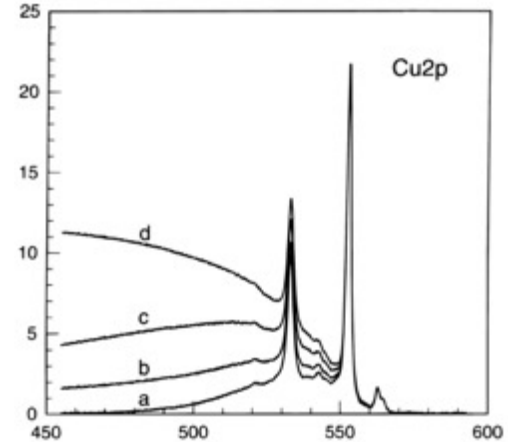
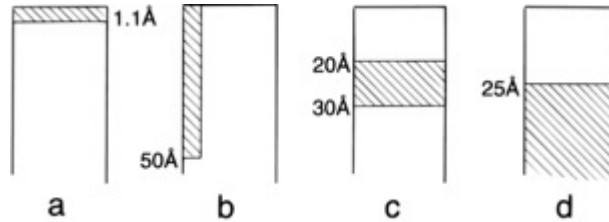
At.-%	C	O	N	F	Si
As received	45.7	26.8	0.0	2.8	24.7
after O <sub>2</sub> plasma	18.5	53.9	0.0	0.0	27.6
Non fouling coating	40.3	34.0	4.3	0.7	20.6

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# How does Morphology influence XPS



spectrum of emitted electrons:

$$J(E, \Omega) = \underbrace{\int dE_0 F(E_0, \Omega) \int ds e^{-i2\pi s(E-E_0)}}_{\text{Intrinsic spectrum at all positions}} \underbrace{\int dx f(x) e^{-x \Sigma(s)/\cos(\theta)}}_{\text{Background}}$$

$$\Sigma(s) = \frac{1}{\lambda} - \int_0^\infty K(T) e^{-sT} dT$$

$f(x) \rightarrow$  sources: concentration of atoms at depth  $x$

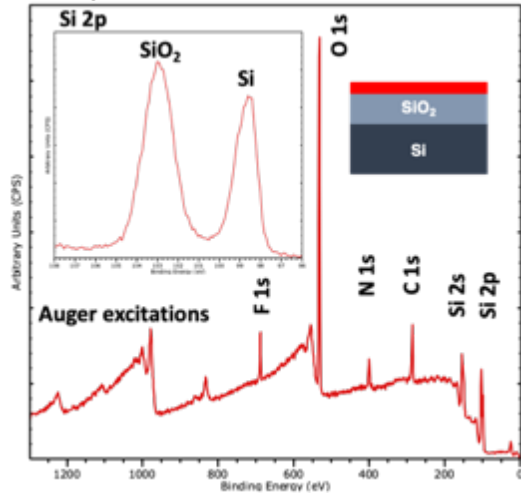
$K(T) \rightarrow$  scatterer: probability that electron will lose energy  $T$





# Roughness of a thin film by XPS

- AziGrip4™ Amine on SiO<sub>2</sub>/Si
- Substrate: Si and O
- Polymer: C, N, O and F

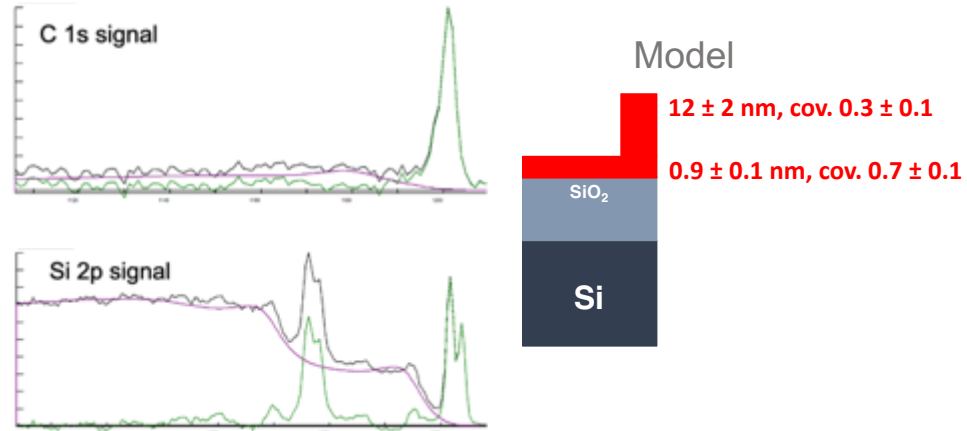


- Film Thickness by VASE

SiO<sub>2</sub> = 3.45 ± 0.04 nm

Polymer = 0.95 ± 0.08 nm → FLAT FILM

- Results from Background Analysis



Surface Morphology of Polymer Chains

knolls/islands of polymer chains  
≈ 10 nm thick  
flat polymer chains  
≈ 1 nm thick

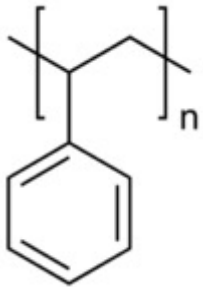


# Polymer Hypergradients

– Hypergradient of two different Polymers

Polystyrene (PS)

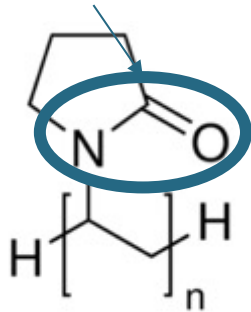
- only aliphatic C



Polyvinylpyrrolidone (PVP)

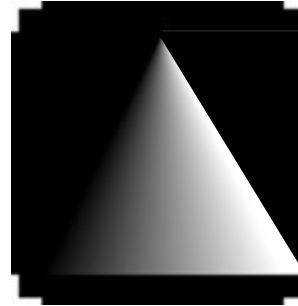
- aliphatic C

- N-C=O

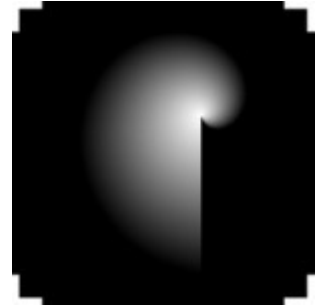


Gradient Patterns

Triangular

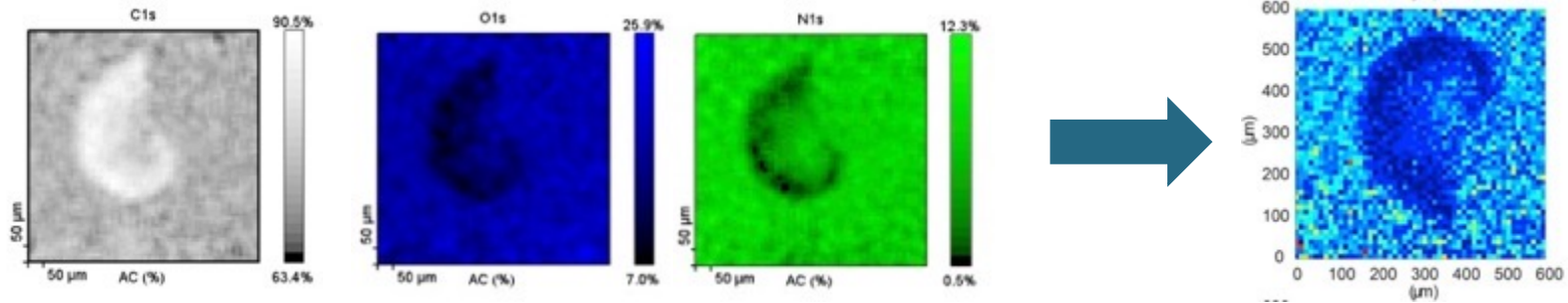


Radial



The difference in chemical composition of both polymers can be used to make hypergradients “visible” via Chemical Imaging

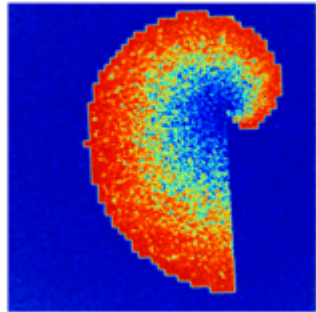
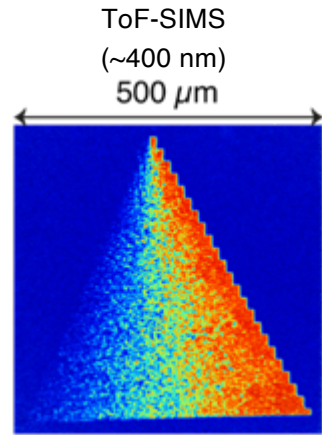
# Image collection with XPS



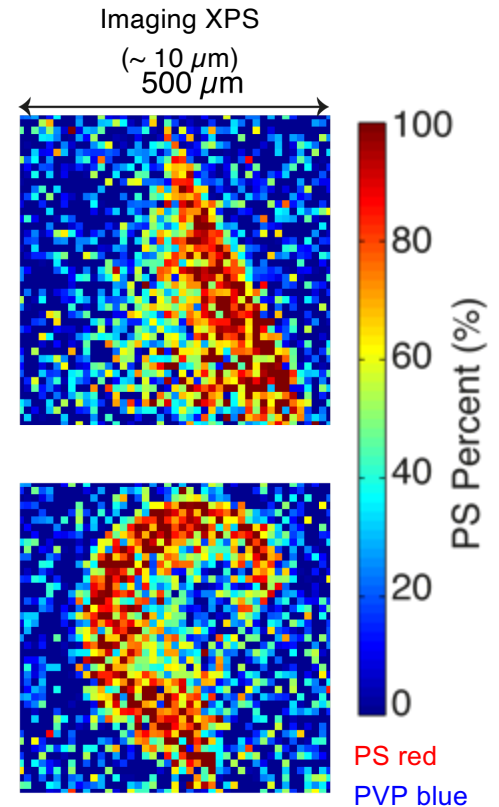
- N/C ration calculated for each pixel
- The percentage of PS was then estimated by assigning the ratio found on pure PVP to 100 % PVP, and N/C=0 to 100 % PS according to:

$$PS_{frac} = 1 - \frac{\left(\frac{N}{C_{frac}} - \frac{N}{C_{PVP}}\right)}{\left(\frac{N}{C_{PS}} - \frac{N}{C_{PVP}}\right)}$$

# Comparison of Imaging Techniques



- Lateral resolution is limited to size of X-ray beam.
- A semi-quantitative answer is possible at each acquired pixel.
- ToF-SIMS alternative for higher resolution images BUT
  - limited in terms of samples
  - quantification hardly possible





# Summary

- Polymers are flexible, versatile applicable materials used in Medical Devices, Diagnostics, Microfluidics and many other applications
- Polymer Thinfilms can be tuned in many ways with versatile functionalities, such as Non-fouling, high lubricity, selective binding, adhesion promotion and many more.
- By choosing the right bonding groups many materials can be treated, such as metals, ceramics, glass and plastics.
- The analysis of polymers can be hindered by various factors such as non-conductivity, carbon=carbon, surface roughness or degasing behavior.
- In XPS these obstacles can be mostly overcome and information on the qualitative and quantitative chemical composition can be obtained.
- With Inelastic Background Analysis the Nanomorphology of substrates and thinfilms can be assessed.
- With Imaging XPS even hypergradients of different polymers can be mapped and quantified.



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