

## Testing Facility for “In-situ” Surface Chemical Analysis

### Key words

Ageing, durability, Electron Spectroscopy for Chemical Analysis (ESCA, XPS), Time of Flight-Secondary Ion Mass Spectroscopy (ToF-SIMS), X-ray Absorption Spectroscopy (XAS), synchrotron radiation, surfaces, polymers, plasma

### Fields of application

Surface chemical analysis of plasma modified or plasma-deposited functional layers and surfaces, of their ageing and durability. Special focus on applications in medical diagnostics and for development of implant (bio-)materials.

### Methodology and instrumentation

Preparation chamber (plasma reactor) connected to surface analytical instrumentation, Electron Spectroscopy for Chemical Analysis (ESCA, XPS), Time of Flight-Secondary Ion Mass Spectroscopy (ToF-SIMS), X-ray Absorption Spectroscopy (XAS)

### Items tested

Organic as well as inorganic films and interfaces

### Quantities / characteristics tested

Chemical composition of solid surfaces: Element surface concentrations > 0.5 atom %, compounds at concentrations > 10 % of a monolayer

### Uncertainty / reliability of results

Element surface concentrations: 1 to 20 % relative uncertainty (depends on element),  
Compounds: 20-50 % relative uncertainty, depending on compound and matrix.

### Qualification and quality assurance

The option of “in-situ” surface chemical analysis by using a plasma reactor directly connected to different high-end options of surface analytical instrumentation (ESCA, ToF-SIMS and XAS) is unique in Germany.

Use of monitored surface analytical instrumentation, calibration of photoelectron spectrometer following ISO, participation and organisation of international inter-laboratory comparisons

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## Further information

The „Testing utility for “in-situ” surface chemical analysis“ enables a comprehensive determination of surface chemistry of the very surface of functional layers avoiding any influence of ambient air after their preparation. This characterisation comprises quantitative analysis of elements and (semi-)quantitative determination of chemical species, especially functional groups. The influence of a exposure to ambient air on freshly deposited samples can be avoided, and, therefore, the ageing and durability of the surface can be chemically studied in detail.

As an example, the investigation of the influence of deposition conditions (plasma process parameters) on the chemistry of plasma polymerised polystyrene films is used to illustrate the application of the „Testing utility for “in-situ” surface chemical analysis“ (cf. Refs. [1, 2]).

Effects of the plasma process parameter duty cycle, plasma power and monomer partial pressure in the reactor chamber on the chemistry of deposited plasma polystyrene films and the ageing of these films were investigated. In detail, ToF-SIMS provided semi-quantitative data on the level of branching and cross-linking. The retention of aromatic rings can be also monitored by this method. ESCA was used to determine quantitatively elemental surface concentrations. The retention of aromatic rings was monitored semi-quantitatively, too. Applying XAS unsaturated carbon species were determined semi-quantitatively. For instance, cross-linkings by substitutions at the aromatic rings were observed by this method.

The investigation of ageing phenomena due to exposure to ambient air by ESCA provided quantitative oxygen uptake data in dependence on the plasma deposition parameters. These results were semi-quantitatively cross-checked by ToF-SIMS at enhanced sensitivity. Different oxygen species are formed due to the auto-oxidation processes inherent to ageing. Carbonyles were semi-quantitatively determined by XAS at high sensitivity. C-OH, C=O and COOH species were quantitatively determined by ESCA. A key species for auto-oxidation is the COO<sup>•</sup> radical. The kinetics of its decay were analysed by ToF-SIMS.

This short example shows how it is possible to obtain comprehensive chemical information for organic films by using the „Testing utility for “in-situ” surface chemical analysis“. The different analytical methods are used complementing one another as well as cross-checking selected results. The reliability of surface analytical results, e.g. the retention of aromatic rings in the given example, can be substantially enhanced by this self consistent approach.

[1] U. Oran, S. Swaraj, J. F. Friedrich and W. E. S. Unger, *Surface analysis of plasma-deposited polymer films, 1: Time of Flight Static Secondary Ion Mass Spectrometry (ToF-SSIMS) of plasma polystyrene before and after exposure to ambient air*, Plasma Processes and Polymers 1 (2004) 123-133.

[2] Swaraj, S.; Oran, U.; Lippitz, A.; Schulze, R.-D.; J. F. Friedrich, Unger, W. E. S., *Surface analysis of plasma-deposited polymer films, 2: Analysis of post-plasma air reacted plasma polymerized styrene by X-ray photoelectron spectroscopy and X-ray absorption spectroscopy*, Plasma Processes and Polymers 1 (2004) 134-140.

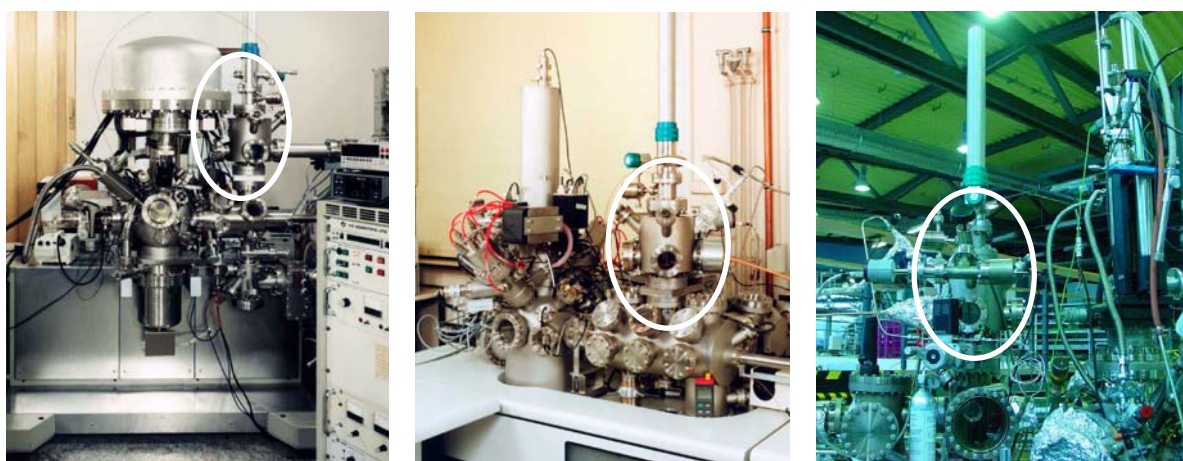


Figure: Plasma reaction chamber (in a white ellipse) directly connected to ESCA- (left), TOF-SIMS- (middle) and X-ray absorption (XAS, right) instruments. The X-ray absorption spectroscopy uses synchrotron radiation provided by BESSY II.