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■ Efficiency of Cleaning Steps

TAS-AN-T1E

Surface cleaning is an integral part in almost all production processes. In particular, cleaning is necessary before coating steps in order to promote optimum adhesion results. Several cleaning methods are common in industry, among others plasma and corona treatments, ultrasonic baths, UV ozone treatments as well as wet cleaning procedures based on water or organic solvents. The efficiency of any cleaning method has to be judged by its performance in two areas: the removal of unwanted material and the avoidance of cleaning debris and residue formation. Modern surface analytical techniques can help to optimise cleaning results in both areas.

Test inks or pens are tools to probe the surface free energy (contact angle). They are easy to use and therefore often applied directly at the production site. However, the contact angle is influenced not only by the overall surface chemistry but also by its topography. Details on the chemical composition cannot be gained. Nevertheless, for the fast control of on-going production processes these methods fulfil a useful purpose.

For a thorough view on the phenomena governing cleaning processes, however, techniques are required which can give details on both, the elemental and the molecular composition of a surface. Here, one can benefit from the specific advantages of Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS), a technique which can sensitively detect atoms and molecules in the uppermost monolayer of a surface. By choosing suited analytical conditions, additionally the lateral distribution of the surface species can be determined with detection limits in the ppm (atoms) and fmol (molecules) range.

In the following, two examples are given demonstrating the capability of ToF-SIMS analysis for the monitoring of cleaning efficiencies.

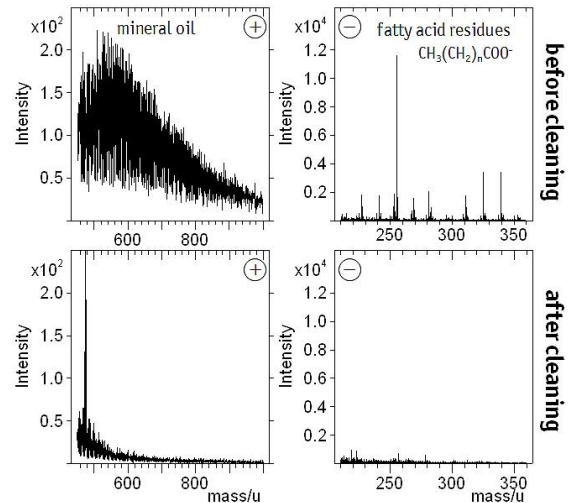


Figure 1: Monitoring of cleaning efficiency by ToF-SIMS; upper row: data before cleaning; bottom row: data after cleaning and rinsing.

Figure 1 shows ToF-SIMS data from metal surfaces. The upper row shows the spectra before cleaning. On the left hand side, positively charged secondary ions are shown whereas the right hand side presents negatively charged secondary ions. Clearly, the surface contamination by a mineral oil and fatty acid residues can be seen which originate from the metal machining. Using ToF-SIMS it is not only possible to determine the average molecular weight of the mineral oil which is related to its viscosity but the various fatty acid residues can be distinguished, too. In the example above, the most prominent residue is palmitate at mass 255 u. After cleaning the peaks of the mineral oil and the fatty acid residues are clearly diminished, proving the success of the cleaning procedure. The secondary ion signals around 485 u (bottom left box) belongs to a vestige of the used detergent.

The use of surface analytical techniques like ToF-SIMS not only allows monitoring the removal of surface contaminants it can also visualise the presence of cleaning remnants. As an example the following figure shows ToF-SIMS images of a Ni surface which was treated using a water-based multi-bath cleaning procedure. All cleaned surfaces showed a bad adhesion behaviour in the following

soldering step. Even the additional use of a plasma cleaning step could not improve the adhesion strength.

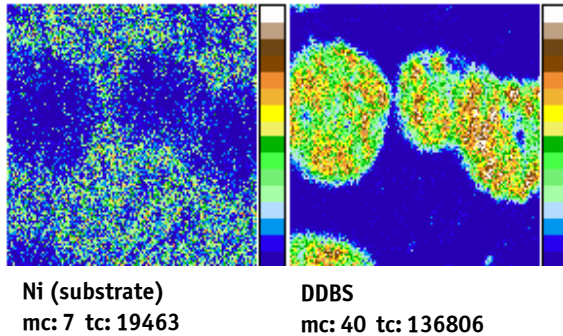


Figure 2: Mass resolved ToF-SIMS images of a Ni surface after cleaning; left: substrate (Ni), right: signals characteristic of dodecylbenzenesulfonic acid (DDBS) (mc: number of counts in the brightest pixel; tc: total number of counts); Field of view: 500x500 μm^2

The lateral distribution of characteristic substrate ions is presented on the left hand side of figure 2. The right hand side shows the lateral distribution of the cleaning agent dodecylbenzenesulfonic acid. Taking into mind that the information in ToF-SIMS is exclusively coming from the uppermost 1-3 monolayers of the surfaces under investigation it can be concluded that the Ni surface is contaminated by spot-like remnants of the cleaning agent with a local coverage in the multilayer range. It therefore is not surprising that the additional plasma treatment was unsuccessful because it could not remove the locally thick cleaning remnants. The problem could be solved, however, by an optimisation of the rinsing steps in the water-based cleaning cascades. The key factor of success in this case was the molecular information from the ToF-SIMS analysis.