

Sample preparation for ToF-SIMS analysis



A major advantage of ToF-SIMS is that there is no need for significant sample preparation



But you need to pay attention to 4 points in order to run a good analysis.

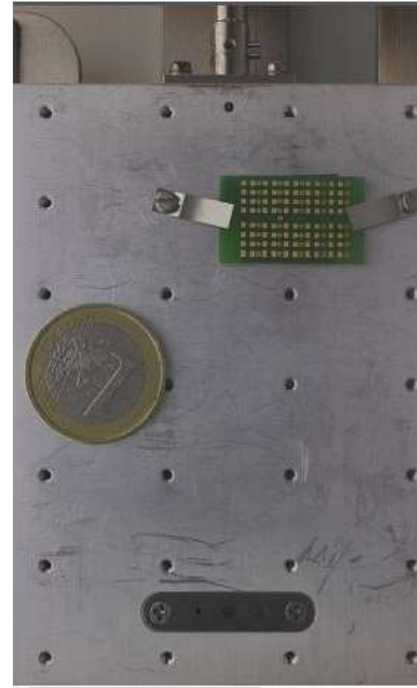
- Sample size (adapted to sample holder and size of the main chamber)
- Flatness of the sample (improve signal and mass resolution)
- Ultra-high vacuum compatible samples
- Sample storage is important to avoid surface contamination (ToF-SIMS is a very sensitive technique ppm – ppb)

Sample size:



Backmount sample holder

Optimally the samples should not exceed 1 x 1 cm²



Topmount sample holder

Samples should not exceed 6 x 8 cm²
Sample height should not exceed 1 cm



*This sample holder is not secured,
danger of crash with the extractor.*

Flatness of the sample:

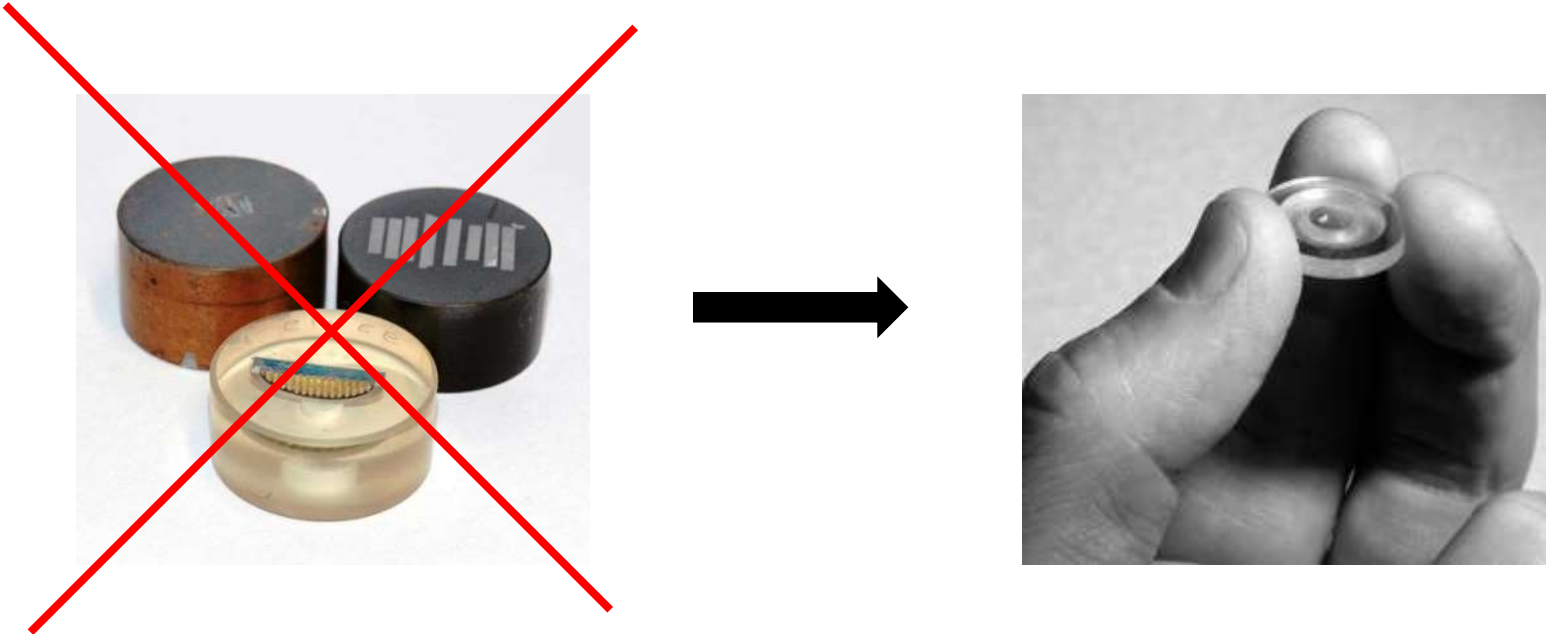


Sample polishing is sometimes essential when the topography is too important.

Excessive topography leads to :

- A lower mass resolution
- A significant decrease in the secondary ions signal.

Ultra-high vacuum compatible samples :



It is often necessary to embed the samples for polishing. The embedding volume should be kept as small as possible because the resin degasses and deteriorates the vacuum in the analysis chamber.

Liquids are not compatible with vacuum and cannot be analyzed by ToF-SIMS. One possibility is to freeze them and keep them frozen during the analysis.

Samples storage

ToF-SIMS is a very sensitive technique. Simply exposing a sample to air will result in the presence of organic contaminants that will be detected during analysis.



Aluminum foil



Glass container



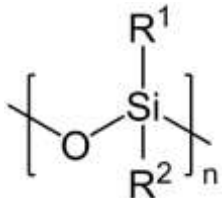
Sample storage under vacuum is the best method.
This type of container allows to avoid as much as possible the contamination or oxidation of a sample.

Samples storage



No plastic bags => High risk of siloxane contamination

Siloxane are very surface active and migrate over the surface and contaminate the sample.

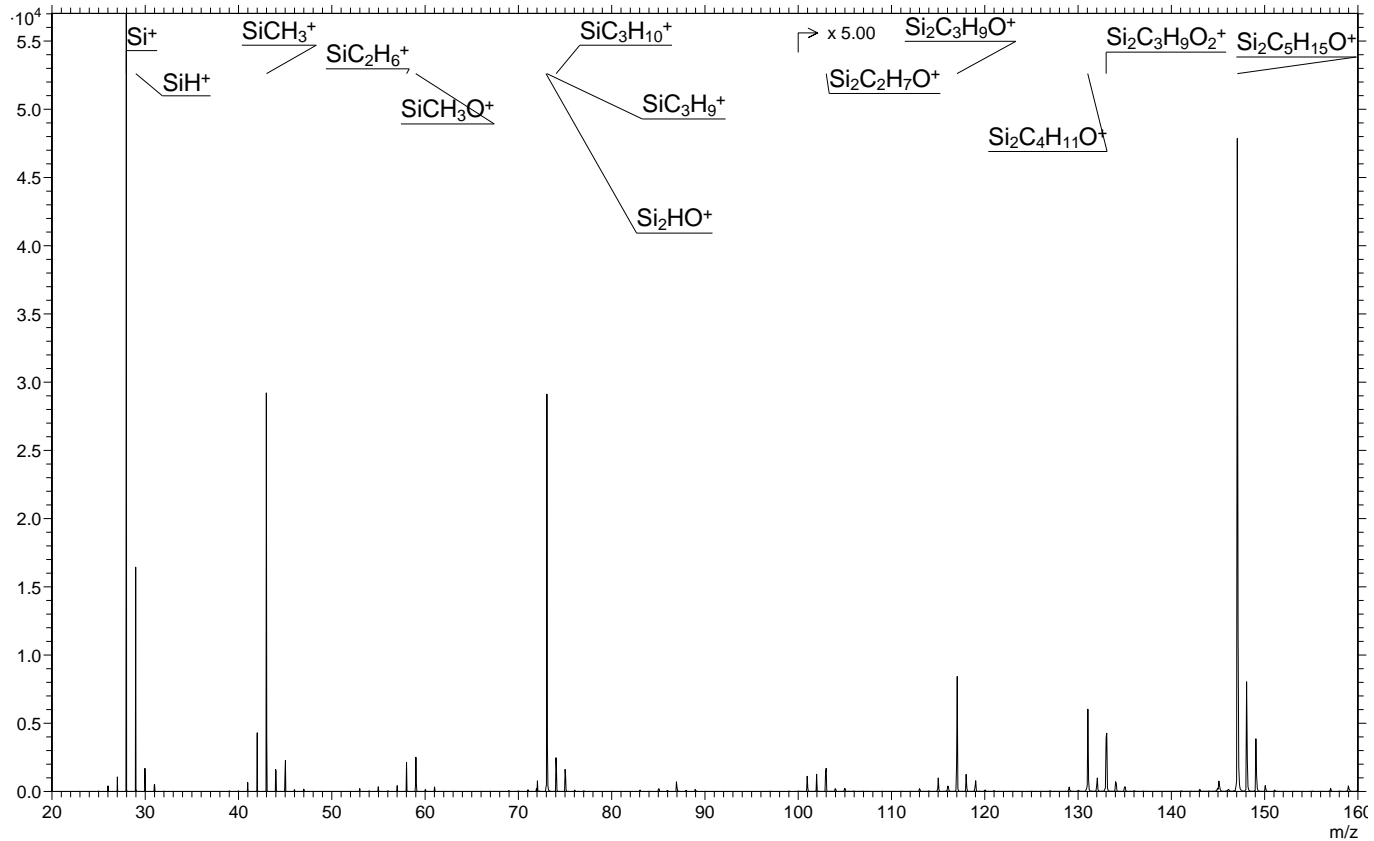


Siloxane structure

Organic groups become attached along the Si-O chain (designated by the symbols R^1 and R^2).

Characteristic spectra of a PDMS contamination

Spectrum of a PDMS contaminated sample in positive mode. Only the main peaks have identified.



When PDMS pollution is too high, the spectra become useless.

Specific preparation for powders

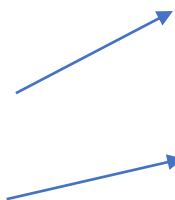


→ Use of adhesive tape is required

But, most tapes, in particular conducting tape (carbon tape) does contain siloxanes as release agent.



Sticky part of Post-ItTM is a siloxane free tape !



Try to flatten the powder as much as possible to limit the topography.

Remove any excess material (loose powder grains) :

- sample up side down
- sample knocking
- Air or Nitrogen stream