

Laser Ablation ICP-MS

Trace analysis without sample preparation

- laser ablation ICP- MS
- no sample preparation
- elemental analysis down to sub-ppm levels
- spatial resolution of 10 μm
- depth resolution of 1 μm

Using a laser small amounts of material are removed from a sample. By an inert gas flow this material is transported into an inductively coupled plasma (ICP), in which an effective temperature of 7000 K results in atomization and ionization of the sampled material. Subsequently, the ions are extracted into a quadrupole mass spectrometer (MS), with which the elemental composition of the material is determined.

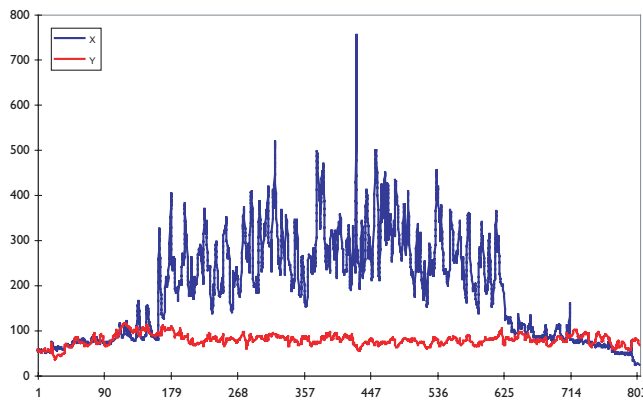


Fig. 2: Concentration distribution of two dopes in an aluminum oxide tube.

Laser ablation is a very clean method of sampling, for which there are no requirements with regard to the structure and composition. This is an advantage for an increasing number of applications.

Ceramics, for instance, are non-conducting and hard to dissolve. This makes analyses by using wet chemical analyses or by most surface analytical techniques difficult. In addition, samples of this type are often structured in the form of tubes etc. that results in non-flat surfaces that are difficult to access.

In figure 1 an ablation pattern of 27 holes (30 µm diameter) is made over a distance of 800 µm on the cross section of a densely sintered aluminum oxide (DGA) tube. This shows that laser ablation can perfectly be used for sampling ceramics. Because of the small amount of material that is sampled with laser ablation, the combination with ICP-MS is an obvious one.

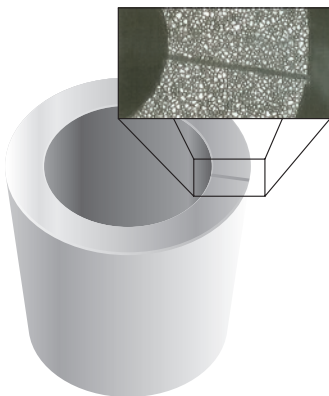


Fig. 1: Ablation pattern on an aluminum oxide tube.

The combination yields detection limits that are between 10 ng/g (rare earth elements) and 1 µg/g (K, Ca, Si) for most elements in the periodic table, without the necessity of sample preparation.

Again this is especially useful for ceramics in which small amounts of additive have a large effect on the characteristics of the material.

Using laser ablation ICP-MS, a preliminary survey analysis was made of the elemental composition of the tube in figure 1 which indicated the presence of two major dopes, named X and Y, at concentrations higher than 25 µg/g. Wet chemical analysis, which could only be done after extensive sample preparation, indicated an average X- and Y- concentration of 180 µg/g and 45 µg/g, respectively. Subsequently, the distribution of these elements over the material was determined using laser ablation ICP-MS.

Figure 2 shows the concentration patterns of X and Y in DGA using the ablation pattern given in figure 1 (27 holes, 300 laser shots per hole). A large difference in distribution of X and Y over the DGA can be observed. Y is distributed relatively evenly, but X shows a very different pattern.

In the bulk, X is located mainly on the borders of the aluminum oxide grains, while close to the surface of the tube the concentrations are much lower as a result of evaporation.

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Technical Note 1
August 2016

Characteristics

Sample type

- solids (bulk, inclusions, thin layers > 1 µm)

Quantitative/qualitative

- semi-quantitative

Lateral resolution

- 10 - 350 µm per shot

Laser wave length

- 193 nm and 266 nm

Depth profile

- possible for e.g. inclusions (depth > 1 mm)

Accuracy

- within a factor 2 for unknown materials

Precision

- depends on sample type

Detection limit

- µg/g in solids for most elements

Sample requirements

- up to 8 cm in diameter or 3 cm in height

Sample quantity

- analysis volume 25 µm³ - 0.1 mm³

Sample (pre)treatment

- none

Destructive/non-destructive

- yes, tiny holes have to be made



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