High-sensitivity Analysis of Alumina Powders by µs-FF-GD-MS and the Thermo Scientific ELEMENT GD MS

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Overview

Purpose: To demonstrate the capabilities of the Thermo Scientific™ ELEMENT™ GD MS for the impurity determination in high purity alumina powders with minimum sample preparation effort.

Methods: µs-FF-GD-MS, Secondary Electrode.

Results: The ELEMENT GD MS in µs-pulsed operation mode provides reproducible and accurate trace metal quantification of high purity alumina powders. The simple sample preparation avoids the risk of contamination, as well as time-consuming dissolution steps and the need for cleanroom conditions. The method results in best matrix sensitivity, and high sample throughput (approximately 2 to 3 samples per hour).

Introduction

Items produced from high-purity Al_2O_3 powders are found in a large variety of consumer and industrial products. With the predicted increased demand for 5N and higher purities, a fast, simple and accurate analytical technique is required to closely control production.

Non-conductive oxidic powders generally require a secondary electrode or binder for being analyzed by
DC-GD-MS. The ELEMENT GD MS equipped with a
µs-pulsed control of the DC-GD ion source makes the use of a secondary electrode a standard application. While other GD-MS approaches, employing secondary electrodes, have been used in the past, they were generally limited by a comparatively low sample throughput of several samples per day.

Methods

Test Specimen

High purity alumina reference materials NMIJ 8006a and 8007a pressed into high purity tantalum.

Mass Spectrometry

The Thermo Scientific™ ELEMENT™ GD MS equipped with a µs-pulsed control of the DC-GD ion source.

TABLE 1. Instrumental settings.

Parameter	Value
Matrix Sensitivity	$2 \cdot 10^9$ cps Al (MR)
Analysis Time	10 min presputter 10 min acquisition
Discharge Voltage	1000 V
Pulse Settings	4 kHz repetition rate 50 µs pulse duration
Anode Parts	High purity graphite

Results

Semi-quantitative multi-element measurements with high sensitivity

A well-characterized high-purity alumina reference sample (NMIJ 8007a) was analyzed repeatedly using the pressing approach shown in Figure 1. Results applying the semiquantitative calibration table used for standard metal DC operation show a good match for the measured concentrations (Table 2).

FIGURE 1. Alumina pellet pressed into high purity tantalum target acting as a secondary electrode.

Finding that this Standard RSF table can be used even when it is applied for this non-conductive material in pulsed mode with a secondary electrode is of importance since typically full scan analyses are required. This triggers the need for quantifying also elements that cannot be readily calibrated.

Due to the high sensitivity of this GD-MS methodology (~$2 \cdot 10^9$ cps for the matrix ion ^{27}Al, Medium Resolution), even at the 0.01 ppm level good precisions are obtained (Table 2).

The sample preparation method is simple, reproducible and clean. The Ta target used is resurfaced by grinding or milling for the following analyses. Si contamination at low ppm level can originate from a SiC grinding step – milling is therefore the preferred method for refurbishing the Ta target. Alternatively, grinding with corundum paper could be applied.

Halogen determination

The determination of halogens has been studied by analyzing the high level CRM 8006a which is certified for its Cl content at 344ppm. The raw ^{35}Cl signal observed yields approximately 400cps per ppm (Figure 2), which is ~20 lower than the typical factor observed for metals. This lower sensitivity is to be expected due to the high ionization potential, and still it is high enough to enable direct Cl determination at or below the single digit ppm level. As an example, the Cl content of the low level CRM NMIJ 8007a has been determined to ~2ppm (Figure 2).

TABLE 2. Semiquantitative results of the high purity Al_2O_3 reference material NMIJ CRM 8007a (all concentration values in ppm). Four repeat analyses included sample preparation were carried out. Values in *italics* are information values.

Element	Measured concentrations	Standard deviation of repeat analysis	Certified concentration
Fe	5.0	0.3	5.01 ± 0.25
Si	19.5	1.3	17.1 ± 0.4
Zr	2.5	0.6	1.80 ± 0.20
B	1.08	0.09	*0.21 ± 0.08*
Ca	2.4	1.0	*0.92 ± 0.14*
Cr	1.15	0.09	*0.84 ± 0.09*
Cu	1.25	0.06	*0.92 ± 0.08*
Mg	3.1	0.2	*2.8 ± 1.1*
Sr	0.025	0.007	*0.022 ± 0.009*
Ti	0.35	0.06	*0.26 ± 0.08*
Th	0.010	0.003	-
U	0.030	0.003	-

Calibration

For full quantification, the RSF table used should be updated based on calibration with reference materials. Especially high IP elements like boron are more efficiently ionized in pulsed mode, making a dedicated RSF table desirable.

To demonstrate this procedure, the two CRM were plotted into a calibration graph showing the linear response of the acquired data (Figure 3). Since only a few elements are certified in both CRM, just two examples are shown. Thus, a one point calibration at higher ppm levels appears to be a valid approach, so that a matrix matched calibration for a wide range of elements can be easily established. For all other elements, the semiquantitative data are being used.

FIGURE 2. Example spectra for chlorine at different concentration levels.

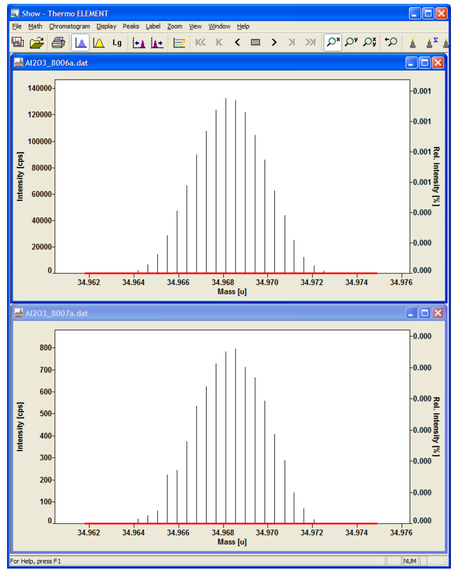


FIGURE 3: Calibration line examples from 2 alumina CRM. Note the logarithmic scale.

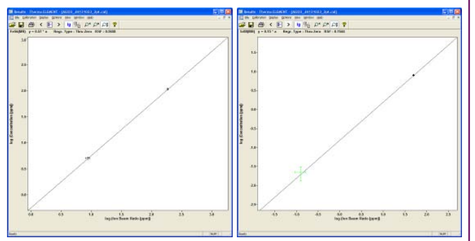
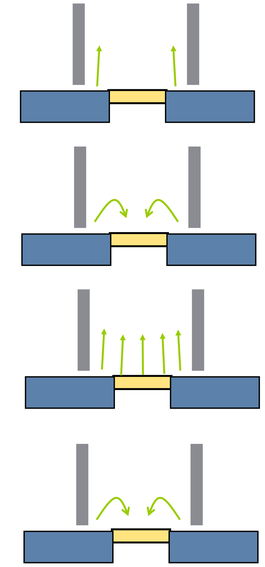


FIGURE 4. Proposed mechanism for alumina analysis using a pulsed discharge with a secondary tantalum electrode.



Conclusion

- The µs-FF-GD-MS technique enables the routine analysis of ppb-level impurities in alumina powders, using high purity tantalum as a secondary electrode. The method proposed results in high matrix sensitivity, higher sample throughput (approximately 2 to 3 samples per hour) and in an easy and fast sample preparation.
- Halogens are accessible for quantification at the ppm level.
- The simplified adjustment of the source and sample components results in an easier user handling process.
- The application field of this new approach is very broad and will likely affect many new research areas.

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