APPLICATION BRIEF 30491

Analysis of alumina powders

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Keywords

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Goal

To demonstrate the capabilities of the Thermo Scientific Element GD Plus µs-Fast-Flow Glow Discharge Mass Spectrometer for high throughput trace metal determination in high purity alumina powders with minimum sample preparation.

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 control production.

Non-conductive oxide powders in general and alumina in particular require harsh conditions for wet chemical dissolution in order to be run on ICP-MS. Direct analysis from the solid provides a cleaner sample preparation method, using a secondary electrode for analyses with DC-GD-MS.

The Thermo Scientific™ Element™ GD Plus GD-MS equipped with a pulsed power supply overcomes the analytical limitations associated with the use of a secondary electrode with high-vacuum GD sources. The µs-pulsed fast-flow source provides state-of-the art solid sample analysis, at a sample throughput of several samples per hour.

Table 1. Instrumental parameters.

Parameter	Value
Matrix intensity	2×10^9 cps Al (MR)
Analysis time	10 min pre-sputter 10 min acquisition
Discharge voltage	1000 V
Pulse settings	~4 kHz repetition rate 50 µs pulse duration
Anode parts	High purity graphite

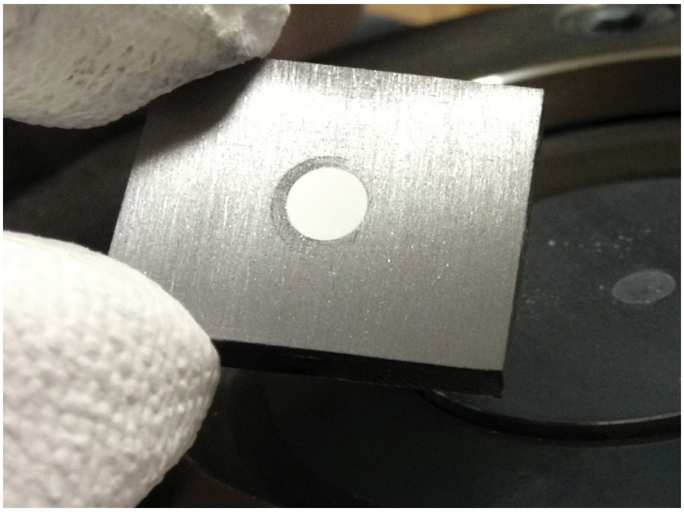

Figure 1. Sample preparation example for pressing non-conductive powder material into a secondary electrode.

- The sample preparation method is simple, reproducible and clean.
- The Ta target used is easily resurfaced by grinding or milling for multiple use (Si contamination at low ppm level can originate from a SiC grinding step, grinding with corundum paper can serve as an alternative). Milling is therefore the preferred method for refurbishing the Ta target.
- Due to the high sensitivity of this GD-MS method (~2 × 10^9 cps for the matrix ion ^{27}Al, Medium Resolution), even at concentration levels as low as 0.01 ppm, good precisions are obtained (Table 1).
- Halogens are accessible for quantification at the ppm level.

Method

For sample preparation, a high purity Tantalum target was equipped with a borehole of approximately 5 mm diameter into which the sample was pressed. The target was placed on a TaW plate, and several tens of milligrams ceramic powder were filled into the borehole and pressed with a TaW pressing pin. The pressure should be adjusted to the kind of powder used. For fine-grained Al_2O_3 samples a pressure of ~0.4 tons, yielded stable and compact pellets, ready to be inserted without further treatment into the Element GD Plus GD-MS sample holder.

Results

- High purity alumina reference materials are reliably analyzed using Ta as a secondary electrode.
- Very good precisions are achieved (Table 2).
- The Standard RSF approach concept is shown to be valid for pulsed mode operation.
- High ionization potential Elements like boron are more efficiently ionized in pulsed mode. Therefore a dedicated RSF table should be applied.
- For the most important Elements, a matrix matched calibration can be easily established (Figure 2).

Table 2. Semi-quantitative results of the high purity Al_2O_3 reference material NMIJ CRM 8007a (all concentration values in µg·g $^{-1}$). Repeat analyses included sample preparation. Values in italics are information values.

Element	Measured conc.	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	—

Conclusion

The Element GD Plus GD-MS in µs-pulsed operation mode is ideally suited for reproducible and accurate trace metal quantification of high purity alumina powders. The simple sample preparation avoids contamination and time-consuming dissolution steps, facilitating a close production control for ensuring highest quality products.

The reference material used is from the National Metrology Institute of Japan, Metrology Management Center, Reference Materials Office, 1-1-1, Umezono, Tsukuba, Ibaraki 305-8563, Japan: http://www.nmij.jp/

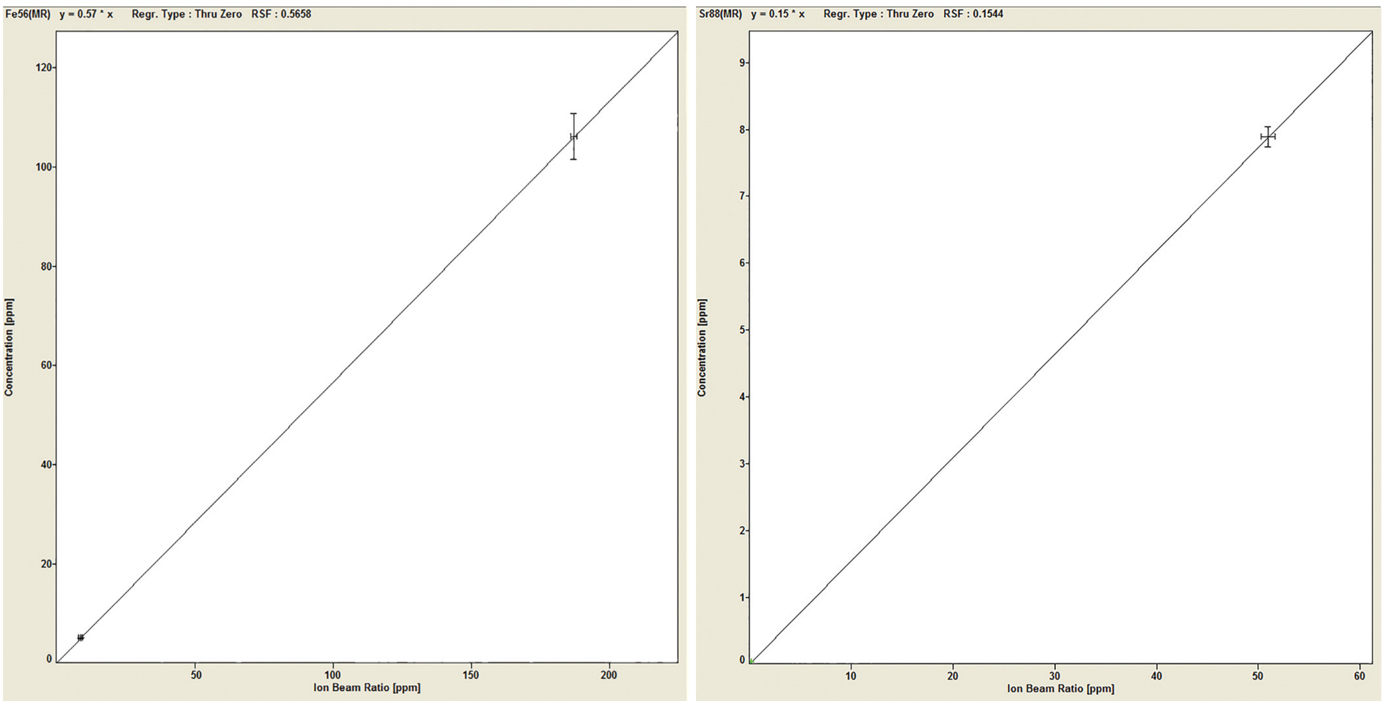

Figure 2. Calibration examples for the alumina reference materials NMIJ CRM 8006a and 8007a. Note the logarithmic scale.

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