CHEMICAL PURITY DETERMINATIONS OF ADVANCED ALUMINA SAMPLES BY ASTRUM GLOW-DISCHARGE MASS SPECTROMETRY

DeAnn Barnhart, Karol Putyera and Glyn Churchill

INTRODUCTION

High purity aluminium oxide has been widely used as fillers for magnetic media, fillers in high performance composite resins, advanced ceramic parts and substrates for electronic components and semiconductor manufacturing equipment for over three decades. Due to the low dielectric constant and high electrical resistivity of this material, the demand for high grade aluminium oxide is only expected to increase in the coming years. As the demand for high grade aluminium oxide increases, so does the demand for accurate characterisation and assessment of material purity. In this application note, the analysis of CRM 8007a alumina powder and a high purity sapphire wafer is explored using the Astrum high resolution GD-MS with a secondary electrode.

Astrum

Experiment

A sample holder was constructed using high purity tantalum to form an electrode by fashioning a groove into the tantalum to hold the aluminium oxide powder. The sample holder was then chemically cleaned and analysed to verify that no impurities were added during the machining process (Table 1). Certified Reference alumina powder NMIJ CRM 8007a was loaded onto the electrode, which was then placed into a sample chuck (Figure 1). The sample was first allowed to be sputter cleaned until the plasma conditions had stabilized (~10 minutes), and then analytical data were collected until at least three consecutive equilibrated readings were reached for each of the selected analytes. The same procedural steps were followed for analysing the optical grade sapphire wafer sample.

Instrumentation

The Astrum is the latest generation of GD-MS instruments developed as the benchmark in GD-MS. The instrument was designed in conjunction with the users of the most widely-used GD-MS, the VG9000. The best design concepts of this system were combined with advances in sample cell design, control electronics and pumping technology to produce a high performance new instrument designed specifically for ultra-trace analysis of impurities. Every effort has been made to ensure that the background level in the instrument is as low as possible, including cryo-cooling of the source.

Discussion

Until recently, analyte material analysed on the Astrum at Evans Analytical Group consisted primarily of pins made from conductive materials. Additionally, it is possible to analyse solid insulating materials with GD-MS by means of a specially prepared sample holder made from highly purified tantalum (Figure 1).

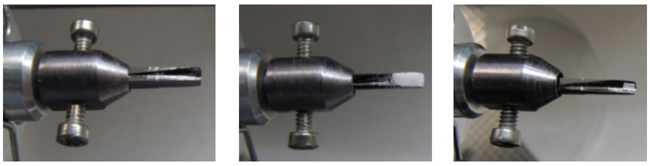

Fig. 1: A high purity tantalum sample holder (left) acts as an electrode in the analysis of alumina powder (center) and sapphire wafer (right).

Prior to every measurement, the impurities of the sample holder were determined to verify that the sample holder was clean and ready for analysis (Table 1).

Element	Isotope	Conc. (ppm wt.)
B	11	< 0.005
Mg	24	< 0.005
Si	28	< 0.001
Ca	44	< 0.01
Ti	48	< 0.001
Cr	52	< 0.002
Fe	56	< 0.005
Cu	63	< 0.01
Zr	90	< 0.01

Table 1: Results from the impurity check on the tantalum sample holder prior to sample analysis.

Results from the CRM alumina powder substrate analysis are depicted in Table 2. The data from the Astrum were highly consistent with the certified reference material values at the sub-ppm level. It should be noted that these GD-MS results have been obtained by using a Standard RSF data set as the starting calibration set for analysing unknown samples. These findings show that with the generalised calibration constants, the GD-MS results are very close to the certified values.

Element	Certified Conc. (ppm wt.)	Conc. AstruM (ppm wt.)
B	0.21 ± 0.08	0.28 ± 0.03
Mg	2.8 ± 1.1	3.0 ± 0.1
Si	17.07 ± 0.38	12.1 ± 0.3
Ca	0.92 ± 0.14	1.6 ± 0.2
Ti	0.26 ± 0.08	0.38 ± 0.06
Cr	0.84 ± 0.09	1.15 ± 0.03
Fe	5.01 ± 0.25	3.4 ± 0.2
Cu	0.92 ± 0.08	0.84 ± 0.08
Sr	0.022 ± 0.009	0.015 ± 0.003
Zr	1.80 ± 0.20	1.5 ± 0.3

Table 2 : A comparison of the concentrations of ten elements measured in high purity alumina powder NMIJ CRM 8007a using the Astrum with a secondary electrode and their corresponding certified values.

Results for the Astrum analysis of row 1-3 elements, transition elements and rare earth elements in high grade sapphire are listed in Tables 3, 4 and 5 respectively. The improvements in detection limits are due to advances in GD-MS technology resulting in increased resolution, better abundance sensitivity and lower background levels. Alternately, a sample holder made from high purity indium or copper may be utilised to significantly lower the detection limits of niobium, molybdenum and tungsten which are normally reported at <50, <20 and <20 ppm wt., respectively.

Conclusions

The analysis of high grade sapphire wafer and NMIJ CRM 8007a alumina powder using a high purity tantalum pin-style sample holder was investigated on the Astrum. Throughout the alumina trials, the Astrum was shown to be a consistent, reliable and versatile high resolution GD-MS instrument readily capable of the analysis of insulative material at the sub-ppm level. The tantalum electrode proved a very effective method in conjunction with the Astrum in delivering highly accurate analytical results from the fine CRM alumina powder and high purity sapphire wafer in accordance with the certified values and data previously reported on the VG9000, respectively. These results illustrate the strength that the Astrum has to expand into the field of trace and ultra-trace characterisation of high grade aluminium-based insulating material analysis.

Element	Isotope	Conc. (ppm wt.)
Li	7	< 0.05
Be	9	< 0.05
B	11	< 0.05
Na	23	< 0.1
Mg	24	< 0.1
Si	28	< 0.1
P	31	< 0.1
S	32	< 0.1

Table 3: Detection limits of row 1 - 3 elements measured in high purity sapphire wafer using the Astrum with a tantalum sample holder.

Element	Isotope	Conc. (ppm wt.)
Sc	45	< 0.01
Ti	48	< 0.1
V	51	< 0.05
Cr	52	< 0.1
Mn	55	< 0.05
Fe	56	< 0.1
Co	59	< 0.05
Ni	60	< 0.1
Cu	63	< 0.5
Zn	68	< 0.5
Y	89	< 0.05
Zr	90	< 0.05
Ru	102	< 0.1
Rh	103	< 0.1
Pd	108	< 0.1
Ag	109	< 0.1
Cd	114	< 0.5
Lu	175	< 0.1
Hf	180	< 0.1
Re	187	< 0.05
Os	189	< 0.05
Ir	191	< 0.05
Pt	194	< 0.1
Hg	202	< 0.5

Table 4: Detection limits of transitional metals measured in high purity sapphire wafer using the Astrum with a tantalum sample holder.

Element	Isotope	Conc. (ppm wt.)
La	139	< 0.05
Ce	140	< 0.05
Pr	141	< 0.05
Nd	144	< 0.05
Sm	152	< 0.1
Eu	153	< 0.05
Gd	158	< 0.05
Tb	159	< 0.05
Dy	164	< 0.05
Ho	165	< 0.05
Er	166	< 0.05
Tm	169	< 0.05
Yb	172	< 0.1

Table 5: Detection limits of rare earth metals measured in high purity sapphire wafer using the Astrum with a tantalum sample holder.