ASTRUM

GD-MS

A COMPARATIVE STUDY OF PIN CELL VERSUS FLAT CELL GEOMETRIES AND THE ANALYSIS OF A HIGH PURITY FOIL AND A THINFOIL USING THE ASTRUM GD-MS

INTRODUCTION

Aluminium is one of the most widely used metals in the world. Its applications extend into the electrical, building, transportation, packaging and aerospace fields. Aluminium is available in varying purities, ranging from alloys used in the construction, automotive and aerospace markets up to high purity 6N (99.9999%) materials used in the semi-conductor industry. Aluminium is derived from alumina (aluminium oxide) which comes from Bauxite, an aluminium ore. At any stage along the transformation process, be it Bauxite ore, alumina or the aluminium end-product, the material can be analysed using the latest generation of GD-MS instrumentation, the Astrum. A notable advantage of the Nu Astrum over some other GD-MS instruments is its ability to utilize both pin and flat cell geometries for sample analysis. With this flexibility, the Astrum can be used to analyse aluminium in virtually every available form, including solid pieces, wires, spheres, slugs and powders. Another advantage is that since the glow discharge source is easily removed and cleaned, matrix memory effects do not present a problem in the analysis of both alloy and high purity materials on the same instrument.

Instrumentation

The Astrum was designed in conjunction with the users of the most widely installed GD-MS, the VG9000. The globally recognized and accepted performance and functional benefits of this traditional system were combined with the latest advances in software, sample cell geometries, electronics and vacuum 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. The low-flow GD source design allows for analysis of bulk materials and powders as well as thin and ultrathin foils (< 1 µm).

The Astrum

Experiment

Aluminium Certified Reference Material (CRM) 122/06 (Brammer) was analysed using both pin cell and flat cell configurations as a comparison study. The analysed samples consisted of a pin approximately 2 x 2 x 22 mm in dimension and a flat piece approximately 2 x 20 x 30 mm, polished with a 240 grit silicon carbide polishing sheet. Additionally, a 6N 250 µm aluminium foil and a 2N (99.1% purity) 2 µm aluminium thinfoil (Alfa Aesar), sample sizes approximately 20 x 20 mm, were analysed by flat cell method. Instrument settings for each analysis are described in Table 1.

Table 1: Instrument settings for the analysis of the aluminium foils and certified reference material

	CRM 122/06	CRM 122/06	250 µm Foil	2 µm Thinfoil
	(pin)	(flat)	(flat)	(flat)
Discharge voltage (kV)	~1	~1	~1	~0.8
Discharge current (mA)	~3.5	2.0	2.0	1.8
Discharge gas flow (sccm)	0.6	0.5	0.5	0.5

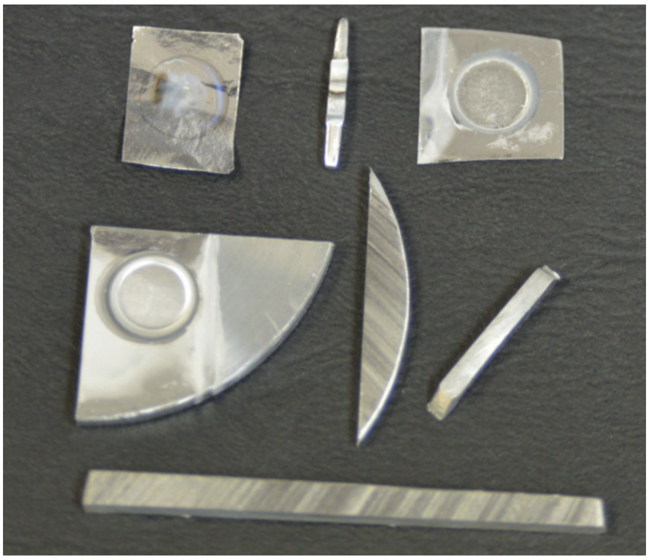
The thinfoil was also analysed at conditions of ~1 kV and ~2mA to corroborate the values collected at the lower conditions. For each analysis, matrix signal strength was $>1x10^{-10}$ A, with the exception of the thinfoil which had a matrix signal strength of $<8x10^{-11}$ A. A resolution of 4000 was used for all acquisitions (R=m/Δm at 10% valley definition). The applied Relative Sensitivity Factors (RSF) were taken from the publication of *Wojciech Vieth and John C.*

Huneke, Relative sensitivity factors in glow discharge mass spectrometry, Spectrochimica Acta Part B: Atomic Spectroscopy, 46 (2), 1991. Although this RSF set was developed for the VG9000, the Astrum was designed such that the RSF data set could also be used for all Astrum data. Each analysis was performed according to the ASTM F1593 Standard Test Method, including determination of detection limits for contaminates.

Discussion

Aluminium CRM 122/06 was analysed using both pin cell and flat cell techniques (Table 2). The pin used for pin cell analysis was analysed three times (Trials 1, 2 and 3), while the piece used for flat cell analysis was analysed twice (Trials 4 and 5).

The results from CRM 122/06 were typical in that the resulting concentrations were within 30% of the certified values. Additionally, the values achieved by pin cell analysis were in good agreement with those obtained from the flat cell method, and the



Aluminium CRM 122/06, 6N 250 µm foil and 2N 2 µm foil, sputtered and unanalysed pieces

Table 2: Results for aluminium CRM 122/06 from both pin cell (Trials 1, 2 and 3) and flat cell (Trials 4 and 5) analysis, concentrations given in parts per million (ppm)

Isotope	Pin cell			Flat cell		Average	Certified	Standard	RSD
	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5		Value	deviation	%
	n=1	*n=1*	*n=1*	*n=1*	*n=1*	*n=5*		*n=5*	*n=5*
^{23}Na	19	18	21	23	19	20	7 - 19	2	8
^{24}Mg	187	174	207	237	215	204	167 - 176	22	11
^{48}Ti	186	137	150	162	158	158	195	16	10
^{51}V	196	150	166	171	165	170	171	15	9
^{52}Cr	243	248	261	287	275	263	197	16	6
^{55}Mn	272	287	269	247	251	265	197	15	6
^{56}Fe	756	691	704	701	707	712	586	23	3
^{58}Ni	222	194	223	239	257	227	201	21	9
^{69}Ga	297	270	266	329	315	295	256	24	8
^{75}As	46	41	43	49	49	46	50	3	7
^{90}Zr	136	98	102	109	104	110	148	14	12
^{98}Mo	128	116	109	105	100	112	91	10	9
^{109}Ag	185	158	151	167	164	165	199	12	7
^{115}In	170	169	146	142	137	153	147	14	9
^{181}Ta	10	10	9	11	10	10	14	1	6
^{184}W	51	39	45	60	57	50	78	8	16

same RSF data set *(Wojciech Vieth and John C. Huneke, 1991)* was able to be applied to both. As alloy material does not require pre-sputter time, total data collection time for each trial was approximately 30 minutes. Results from the analysis of a high purity 6N 250 μm aluminium foil using the flat cell approach are displayed in Table 3.

Table 3: Results for flat cell analysis of a high purity 6N 250 μm aluminium foil

Isotope	Concentration	Isotope	Concentration	Isotope	Concentration
	ppb		*ppb*		*ppb*
^{7}Li	< 5	^{59}Co	< 2	^{128}Te	< 20
^{9}Be	< 5	^{63}Cu	150	^{139}La	< 1
^{11}B	< 5	^{68}Zn	< 50	^{140}Ce	< 1
^{23}Na	< 5	^{69}Ga	< 20	^{141}Pr	< 1
^{24}Mg	90	^{75}As	< 5	^{146}Nd	< 5
^{28}Si	140	^{89}Y	< 1	^{178}Hf	< 5
^{31}P	< 10	^{90}Zr	< 5	^{184}W	< 10
^{32}S	< 20	^{93}Nb	< 2	^{187}Re	< 5
^{44}Ca	< 50	^{98}Mo	< 10	^{193}Ir	< 5
^{48}Ti	80	^{102}Ru	< 10	^{195}Pt	< 20
^{51}V	50	^{106}Pd	< 20	^{205}Tl	< 20
^{52}Cr	70	^{109}Ag	< 20	^{208}Pb	< 10
^{55}Mn	20	^{115}In	< 10	^{209}Bi	< 10
^{56}Fe	< 10	^{119}Sn	< 50	^{232}Th	< 1
^{58}Ni	< 5	^{121}Sb	< 10	^{238}U	< 1

Results from the analysis of the 6N 250 μm aluminium foil showed trace contaminants at the ppb and sub-ppb level verifying the proclaimed purity of the 6N foil. As well as bulk materials, the Astrum is able to analyse thin and ultrathin foils due to the low-flow GD source of the instrument. As this source design allows for a sputter rate of ~10 μm/hr for aluminium, ample time is provided to acquire meaningful and reproducible data for a 2 μm aluminium thinfoil. Results from the analysis of a 2 μm aluminium thinfoil, 99.1% purity, are listed in Table 4.

To ensure at least three stable values for each isotope, the results from the 2 μm aluminium thinfoil were achieved using two run files, one with lower mass elements while the other containing the higher mass elements, the total data collection time being approximately 20 minutes. The thinfoil was first analysed with lower conditions of 0.8 kV and 1.8 mA, and then the results were confirmed using normal running conditions of 1.0 kV and 2.0 mA.

Table 4: Results for flat cell analysis of a 2N 2 μm aluminium thinfoil

Isotope	Concentration	Isotope	Concentration
	ppm		*ppm*
^{7}Li	< 0.01	^{63}Cu	400
^{9}Be	< 0.05	^{68}Zn	700
^{11}B	0.5	^{90}Zr	10
^{23}Na	< 0.5	^{98}Mo	2
^{24}Mg	2.6	^{109}Ag	2
^{28}Si	1700	^{115}In	< 0.1
^{31}P	1.4	^{119}Sn	2
^{32}S	< 0.5	^{121}Sb	2
^{44}Ca	< 0.5	^{139}La	< 0.05
^{48}Ti	130	^{140}Ce	< 0.05
^{51}V	30	^{184}W	< 0.5
^{52}Cr	30	^{195}Pt	< 0.05
^{55}Mn	100	^{208}Pb	90
^{56}Fe	4000	^{232}Th	0.02
^{58}Ni	50	^{238}U	0.4
^{59}Co	5		

Conclusions

To compare pin and flat cell techniques on the Astrum, Certified Reference Material aluminium 122/06 (Brammer) was analysed using both methods. The data achieved by pin and flat cell analysis were found to be in good agreement with each other as well as with the certified values. Conveniently, the same RSF data set was able to be used for both the pin and the flat cell data. Although this RSF set was developed for the VG9000 *(Wojciech Vieth and John C. Huneke, 1991)*, the Astrum was designed such that the same historical VG9000 RSF data set can also be applied to Astrum data. A 6N 250 μm aluminium foil was also evaluated by the flat cell approach. This high purity foil demonstrated the ability of the Astrum to achieve detection limits for trace contaminants at the ppb and sub-ppb level. Additionally, a 2 μm aluminium thinfoil was analysed by flat cell methods showing that the low-flow GD source design allows even a thinfoil of 2 μm to be successfully evaluated on the Astrum. The results presented here showcase the versatility of the Astrum. Whether a sample is an alloy, a high purity material or a thinfilm, accurate and reproducible results can be achieved quickly and efficiently on the Astrum.