

Improved SS346A Ni Superalloy Analysis with the Astrum Swift

Keywords

GD-MS, Ni Alloys, Superalloys, Inconel, High Throughput, Static Discharge, Low Flow.

Introduction

The Astrum GD-MS (Glow Discharge Mass Spectrometer) is widely recognised as the gold standard for high purity materials analysis, with a superior user experience and low cost of ownership. Thanks to its low flow DC (static) GD ion source, the Astrum is the instrument of choice when ultimate detection limits, low gas background and flexible sample geometry are needed. However, compared to high purity materials, alloys & lower purity materials analysis places additional demands on analytical instrumentation: precision and total throughput are often of foremost importance.

The development of the Astrum Swift is a step forward for GD-MS, significantly reducing analysis times whilst maintaining the established benefits the Astrum is regarded for. The ease and stability of tuning of the DC GD source combined with shorter integration times, faster magnet circuit & detector electronics and next generation CoDaq software optimises the Astrum for alloys & lower purity materials analysis, where total throughput is key to success.

In this technical note, pin & flat Ni Inconel 100 samples were analysed using the Astrum Swift to compare precision at different analysis times to highlight the improvements made from the Astrum ES.

Method

Two BAS SS346A Ni Inconel 100 CRM samples were prepared, one pin (21 x ~3mm) and one flat (~25 x 25 x 4mm). Both samples were cut to shape then ground using a 240 grit SiC grinding paper and cleaned with deionised water then acetone before loading into sample holders.

Utilising pulsed cryo-cooling, the cells were cooled to -160°C for both samples. Once the glow was established, the source tuning parameters were optimised for maximum signal intensity and 4000 resolving power. Glow discharge was tuned to standard running conditions for each sample type (1kV, 3mA pin, 2mA flat) to maintain standard RSF validity. Ar glow gas flow was regulated by the Astrum Swift Autogas control system to maintain current and voltage. Both samples were allowed to pre-sputter for 10 minutes prior to analysis. Analysis conditions are given in Table 1.

Table 1: Analysis conditions for both pin and flat samples.

Analysis Conditions		
Sample Type	Pin	Flat
Glow I	3mA	2mA
Glow V	1000V	
Ar Flow	~1.33 ml/min	~0.59 ml/min
Isotopes	78	
Steps	80	
Matrix I Avg. (Ni58)	1.04E-09A	2.32E-10A
Consumables	None	
Cell Temperature	-160°C	
Resolving Power	4000	
Number of Repeats	5	

Analyses were performed at 2 different integration times: one mimicking the Astrum ES and one being the optimum integration time for high throughput with the Astrum Swift. Integration times are listed along with measured analysis times in Table 2 and precision is presented for pin and flat samples in Table 3. Note that the minimum integration time with the current Astrum Swift faraday preamplifier design is 20ms, and 1ms for the IC. Analyses were performed sequentially and datafiles were processed using the Astrum Data Analysis software application.

Table 2: Integration times and total analysis times used.

Integration Time Faraday (ms)	Integration Time IC (ms)	Time Per Repeat (min)	Time Per 5 Repeats (min)	Full Repeats in 30 mins
80	80	10:20	51:40	2
20	5	02:38	13:10	11

Results

Table 3: Precision comparison of pin & flat sample data for 80/80ms Faraday/IC integrations and 20/5ms Faraday/IC integrations, for the 8 matrix elements and 13 trace elements given in the BAS SS346A CRM certificate of analysis. Matrix elements are in bold.

Element	Certified Value (ppm)	PIN		FLAT	
		RSD % Integration Times (F/IC)		RSD % Integration Times (F/IC)	
		80/80 ms	20/5 ms	80/80 ms	20/5 ms
C12	1,500	2.05	2.66	8.90	5.50
Mg24	130	3.07	1.34	5.21	7.75
Al27	55,000	1.96	1.03	5.60	6.46
Ti48	50,000	1.73	2.89	2.75	2.50
V51	10,000	1.50	2.17	3.88	1.79
Cr52	100,000	1.72	1.18	2.26	1.73
Ni58	600,000	0.64	0.50	1.45	0.99
Co59	150,000	1.14	1.17	3.04	2.26
Zn66	28.8	2.16	3.11	56.00	7.67
Ga69	49.6	1.21	1.84	2.03	6.01
As75	50.4	1.82	1.77	3.36	3.12
Se77	5.7	5.29	3.45	3.66	13.36
Mo96	30,000	1.42	1.21	3.28	3.51
Ag107	42.5	1.38	2.51	12.86	2.96
Cd114	0.37	1.96	4.67	2.64	8.75
Sn119	93	1.56	1.46	3.25	5.12
Sb121	45	1.44	1.41	4.04	3.93
Te130	9.3	8.03	3.71	2.05	12.51
Tl205	1.9	0.83	12.37	3.81	8.29
Pb208	22.2	1.07	2.29	7.96	11.78
Bi209	10.3	2.13	2.24	4.54	7.79

- As it can be seen in Figure 1 the Astrum Swift shows a marked reduction in analysis times when compared to the Astrum ES, greatly increasing sample throughput. The improvements in efficiency and magnet electronics reduce repeat times to 10m 38s, a reduction of 1m 18s from the Astrum ES (standard 78 isotope analysis with 80ms integration times).
- With development to the detector electronics the time for one repeat is reduced to 2m 20s, with 20ms Faraday and 5ms IC integration times (standard 78 isotope analysis with 80ms integration times).
- This study demonstrates that high precision is achievable at high throughput even with short integration times of 5ms using the Astrum Swift. 20/5ms was chosen for this study as it gives the best balance between high precision and short integration times. However, for applications where high throughput is of greater importance, integration times as short as 1ms are possible.

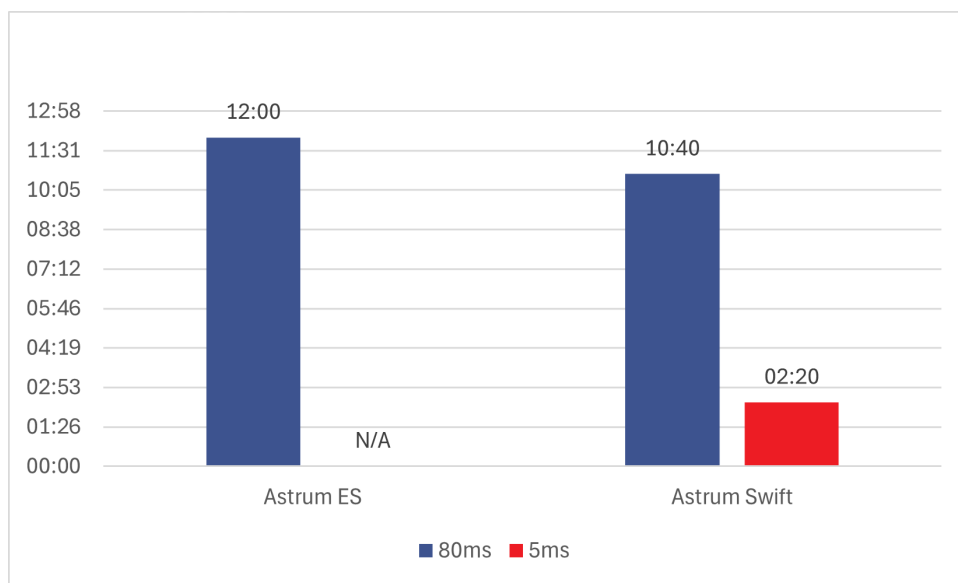


Figure 1: Astrum ES vs Astrum Swift total repeat analysis times, for a typical 78 isotope full mass range method.

Discussion

The Astrum Swift combines the unique attributes of the Astrum with faster throughput, making it the ultimate tool for alloys & lower purity materials analysis. The Astrum Swift's low flow DC source brings multiple benefits that are unique to this source design: glow gas consumption is kept to a minimum, with typical gas flows ranging from 0.2 – 1.0 ml/min. The major benefit of the low flow design is inherent stability and ease of tuning – with no daily or inter-sample calibration necessary. Tuning files can be shared between similar samples whilst maintaining RSF validity when standard glow conditions are maintained.

Solid sample analysis with the Astrum Swift is ideal for high performance alloys such as Inconel. Minimal sample preparation removes complexity from the workflow, and eliminates the risk of contamination during sample digestion, commonly encountered with liquid sampling techniques.

The Astrum Swift was designed with a comprehensive approach to workflow efficiency, from hardware design to software architecture. The sample introduction system with removable sample holders allows multiple samples to be prepared prior to analysis, and sample changeover, including pumping times, takes <2 minutes. The Astrum Swift features a reusable tantalum and ceramic source with zero consumable components, which can be acid cleaned for minimal memory effects.

CoDaq software enhances efficiency with new features such as high-power pre-sputtering, autotune and automatic detector calibration, to minimise user intervention time. The modern approach to user interface design keeps things intuitive for reduced training times. Fully customisable layout and user levels allow simplified control over standard operating procedures.

Summary

The Astrum Swift provides a comprehensive analytical solution for the challenges of alloys & lower purity materials analysis. Designed to fit seamlessly into any workflow, the Astrum Swift brings the benefits of a low flow DC GD-MS to high throughput, high precision industries.

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