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APPLICATION BRIEF 30490

High precision nickel alloy analysis

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Keywords

GD-MS, Nickel Alloys, modulated discharge, Pulsed

Goal

To achieve superior performance of the Thermo Scientific Element GD Plus GD-MS for the analysis of Ni alloys in modulated mode, compared to the bulk analysis of conductive metals with continuous DC GD-MS.

Introduction

The Thermo Scientific™ Element™ GD GD-MS is used and well accepted for trace metal determination in Ni alloys controlled by aerospace regulations. With the upgrade to a modulated supply of the GD source, the precision achievable on the Thermo Scientific™ Element™ GD Plus GD-MS needs to be verified. The accuracy is dominated by the calibration materials used and is equivalent to continuous DC operation.

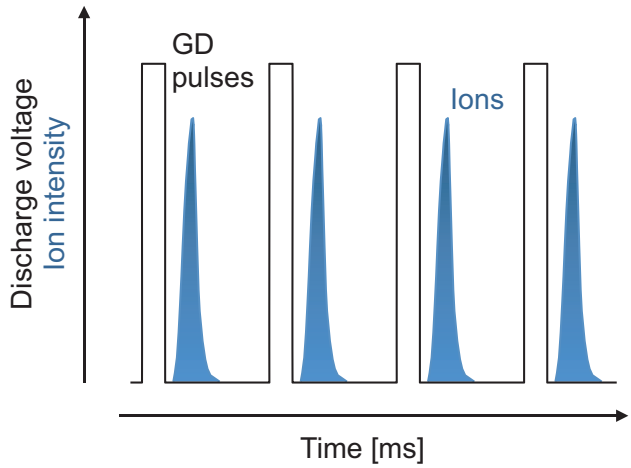

Figure 1. Schematics of pulsed discharge operation.

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Method

The Certified Reference Material (CRM) BAS346A is a nickel superalloy (IN100) with a well characterized set of trace metals important for materials used in the aerospace industry. Therefore this CRM has been used exemplarily to investigate the precision achievable in routine GD-MS quality control tasks.

Repeat analyses were performed including sample preparation by wet grinding the sample on SiC paper grit 80, followed by a washing step with de-ionized water. As a finish, the sample surface was soaked with iso-propanole and blown dry in a nitrogen stream.

Table 1. Instrumental parameters.

Parameter	Value
Discharge voltage	800 V
Pulse frequency	4 kHz
Pulse duration	40 µs
Average discharge current	~15 mA
Matrix intensity	~$1 \cdot 10^{10}$ cps (MR)
Presputter time	6 min
Acquisition time	6 min
Anode consumables	Stainless steel

Table 2. Precision data of repeat analysis of CRM BAS346A.

Element	Isotope (Resolution)	Certified value [µg·g^{-1}]	RSD of all 16 spots	RSD of 6 spots with flow tube and cap exchange	RSD of 4 spots with cap exchange	RSD of 6 spots without any part exchanged
B	11 (MR)	~200	4.2%	4.0%	1.3%	1.2%
C	12 (MR)	~1500	3.2%	4.3%	2.9%	0.8%
Mg	24 (MR)	130 ± 5%	1.2%	1.1%	0.7%	1.2%
Ca	44 (MR)	~20	8%	11%	6%	6%
Zn	66 (MR)	28.8 ± 5%	2.5%	2.3%	1.1%	1.1%
Ga	69 (MR)	49.6 ± 4%	1.5%	1.8%	0.7%	0.9%
As	75 (MR)	50.4 ± 5%	1.2%	0.6%	1.0%	1.1%
Se	82 (MR)	5.7 ± 14%	10%	13%	8%	9%
Ag	107 (MR)	42.5 ± 2%	2.4%	2.0%	1.6%	0.7%
Cd	111 (MR)	0.37 ± 11%	9%	9%	9%	11%
In	115 (MR)	~20	1.6%	1.6%	1.1%	1.1%
Sn	119 (MR)	93 ± 9%	0.9%	1.0%	1.0%	0.9%
Sb	121 (MR)	45 ± 9%	1.2%	0.9%	1.1%	1.2%
Te	130 (MR)	9.3 ± 9%	7.7%	10.6%	5.5%	3.8%
Tl	205 (MR)	1.9 ± 16%	3.0%	3.4%	0.7%	3.3%
Pb	208 (MR)	22.2 ± 5%	1.4%	1.7%	0.6%	1.1%
Bi	209 (MR)	10.3 ± 7%	2.4%	3.0%	1.1%	2.4%

Results

- The results in Table 2 indicate excellent reproducibility for 16 repeat runs including sample preparation.
- Presputter and acquisition times in modulated (pulsed) mode are comparable to continuous modulated operation.
- Best precisions are observed when no source parts are exchanged. This allows for extended analysis (>2 h sputter time, equivalent to approximately 10 sample runs) of similar sample types.
- The typical procedure of changing the anode cap does not significantly impact the precision.
- The exchange of the flow tube shows a visible impact, but still gives precisions similar or better than the reference values itself. As the flow tube receives only small amounts of redeposited sample material, it is typically exchanged on a daily basis.
- A long-term test run on one spot without sample change showed that even after 4 hours the measured concentrations are within a 10% limit. About half of this variation originates from the deep crater eroded, which is never achieved in routine work with ~10–15 minutes sputter time per sample.
- The methodology shown here is subject to ongoing and further optimization, depending on individual lab requirements.

Conclusion

The Element GD Plus GD-MS offers a solution for highly precise trace metal determination in nickel super alloys. Its much lower sputter rate at a similar level of intensity compared to the non-modulated mode yields an excellent spot-to-spot as well as per-spot precision.

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AB30490-EN 1118S