

## SRS 3400

# ANALYSIS OF NICKEL SUPER-ALLOYS

### Background

For many years X-ray fluorescence spectrometry (XRF) has been used as a routine laboratory tool for the analysis of a wide range of metals used in the production of Super-Alloys.

These alloys have a wide variety of uses including the manufacture of turbine blades and assorted marine applications.

Accurate determination of the alloy's component metals is essential to ensure the quality of the finished product. Speed of analysis is also extremely critical as often the processes operating in the production furnace are monitored and accessed using data generated by the XRF spectrometer.

Super-Alloys are principally based on a core metal of either Iron, Cobalt or Nickel with minor and trace amounts of other metals added. This application note deals with Nickel based products and the various alloy groups that fall within this category.

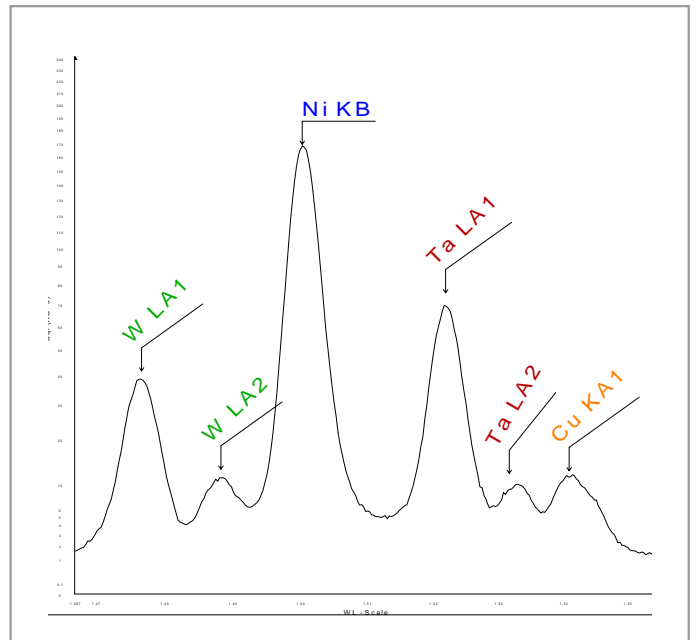


Figure 1: Typical spectrum of a Ni-Alloy

### Instrumental

The Bruker AXS SRS 3400 (shown below) is a high performance sequential wavelength-dispersive XRF spectrometer. It is fitted with a 4 kW end-window Rhodium X-ray tube, up to ten primary beam filters, up to four collimators and up to eight analysing crystals.



Flow and scintillation counters can be used either independently or in tandem. All of this is coupled with Bruker's reliable goniometer technology.

A wide range of sample changers is available, from 1 to 200 positions, and these can be directly linked into automated sample handling systems.

### Software

Perhaps the most outstanding feature of the SRS 3400 analytical system is the inclusion of the SPECTRA<sup>plus</sup> software package. This is a fully interactive program that uses the Windows NT platform for maximum speed and stability.

It comes complete with a standardless analysis routine, a rapid data retrieval system and full networking capability.

### Calibration and Results

An eight minute measurement method was developed with care taken to select appropriate peak and background positions.

The instrument was calibrated using forty-three high quality nickel alloy standards. The standard set was composed of many alloy types with varying concentrations of Chromium, Cobalt, Molybdenum, Tantalum and Tungsten.

Eighteen analytes were calibrated in total. An excellent set of regression lines was produced. Figure 2 gives the Cobalt line as an example.

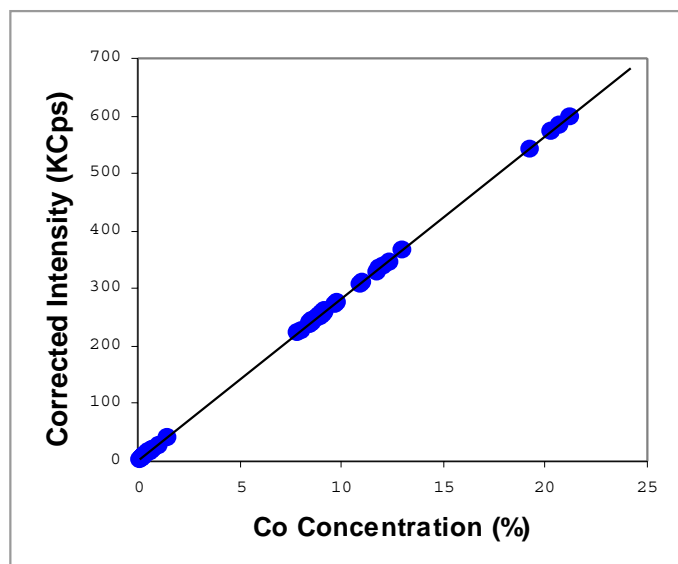


Figure 2: Cobalt calibration curve

A full set of calibration results is given in table 1. It clearly demonstrates the high level of accuracy attainable - even with alloys of markedly different compositions.

This accuracy is only achieved with the "variable alphas" matrix correction model, which is an integral part of the SPECTRA<sup>plus</sup> software package.

This model is based on the philosophy of individual matrix correction for every standard and unknown sample that is analysed. A single calibration line for a wide range of alloys is then possible.

This ends the need for the traditional empirical alpha correction programs, which were prone to analytical errors when routine samples of a slightly different composition were measured.

Table 1: Calibration details

Analyte	Range of Conc. (%)	Calibration Accuracy (%)	Precision R.S.D. (%)
Al	0.15 - 6.90	0.07	0.17
Si	0.10 - 0.58	0.03	0.89
P	0.003 - 0.210	0.029	1.67
Ti	0.08 - 4.36	0.03	< 0.05
V	0.10 - 0.70	0.01	1.14
Cr	5.26 - 31.91	0.14	0.20
Mn	0.06 - 0.55	0.01	0.38
Fe	0.04 - 1.41	0.02	0.22
Co	0.14 - 21.15	0.04	0.12
Ni	Balance	not analysed	0.10
Cu	0.02 - 0.49	0.01	0.61
Y	0.012 - 0.089	0.004	< 0.10
Zr	0.020 - 0.210	0.002	0.75
Nb	0.04 - 2.83	0.02	0.98
Mo	0.12 - 10.09	0.07	0.35
Hf	0.56 - 1.70	0.01	< 0.10
Ta	0.11 - 8.16	0.04	0.25
W	0.05 - 10.87	0.07	0.26

The precision data (R.S.D.) quoted was collected over a twelve hour period incorporating seventy five analyses of the described method.

#### Conclusion

This application note has summarised the outstanding performance of the Bruker AXS SRS 3400 in the field of Nickel Super-Alloy analysis.

By utilising the unique features of SPECTRA<sup>plus</sup> a whole range of alloy types can be plotted on single calibration lines with superb levels of accuracy.

The instrumental precision data given has demonstrated the excellent stability of the spectrometer.

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