MEASUREMENT OF HIGH PURITY METALS USING THE NU ASTRUM GD-MS: THE EFFECT OF MEMORY

INTRODUCTION

There is an increasing demand for ultra high purity metals in today's society, driven by the demands of the semiconductor industry and performance-driven manufacturing sectors such as the aerospace industry. Metals used for these applications must be certified to have impurity contents below certain thresholds. Typically these impurities are at the sub mg/Kg level and there are few techniques capable of measuring such low levels in a solid metal matrix. The most widely used technique for ultra high purity metals analysis is glow discharge mass spectrometry (GD-MS). However, a potential issue with some GD-MS instrumentation is that when a metal is analysed, it leaves a residual "memory" of that metal, interfering with the accuracy of subsequent measurements. In day to day operation this can become a problem, for example when alternating the analysis between one matrix element and another. In this application note, we report on a simple experiment that demonstrated the minimal memory effect of the Nu Astrum GD-MS when a simple cleaning step was followed.

Instrumentation

The Nu Astrum is the latest generation of GD-MS instruments developed to be 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.

Experiment

In order to test the effect that sample memory can have on the Nu Astrum, a series of measurements were made. Firstly, an ultra high purity Cu sample (sample 1) was obtained from a commercial partner of Nu Instruments. Measurements were made of this sample to quantify the composition. Although over 70 elements were measured, for brevity we have chosen ten that best represent the sample composition. The concentration (in mg/Kg) of each element measured is given in Table 1, along with the specific isotope(s) measured. Note that relative sensitivity factors (RSF) are not given as they are beyond the scope of this article. Once the sample had been analysed, the glow discharge cell (including sample holder but not lenses or other ion optic components) was removed from the instrument, cleaned in concentrated HNO_3 and then refitted, a process completed in a couple of hours. A second sample was loaded, this time a Zn sample (sample 2). This sample was analysed for two hours and was then removed and the cleaning process repeated. Finally, the Cu sample (sample 1) was reloaded and the analysis repeated. The data from this analysis is shown in Table 2.

Discussion

The data shown in Table 1 indicates that the Nu Astrum is capable of detecting trace amounts of impurities in a Cu matrix to very low ppb levels. The limits of detection of the instrument are broadly similar to the VG9000 and significantly lower in some cases. A comparison of the data in Tables 1 and 2 gives an impression of the Astrum's susceptibility to memory effects. It can be seen that the trace elements are measured at approximately the same concentration levels both before and after the analysis of the Zn sample. Most convincingly, the trace Zn concentrations measured in the Cu sample are the same before and after the Zn measurement, without recourse to cleaning of extraction optics. This indicates that a short clean of the glow discharge cell is all that is required to return the instrumental backgrounds to the original level when changing the sample matrix.

Element	Isotopes Measured	mg/Kg
Mg	24	0.0001
Al	27	0.0054
Cr	52	0.0008
Mn	55	0.0003
Fe	56	0.0033
Ni	58,60	0.0010
Zn	68	0.0228
As	75	0.0052
Ag	107,109	0.0018
Pb	207	0.0026

Table 1: *Concentrations of ten elements measured in a high purity Cu sample using the Nu Instruments Astrum*

Element	Isotopes Measured	mg/Kg
Mg	24	0.0006
Al	27	0.0026
Cr	52	0.0008
Mn	55	0.0005
Fe	56	0.0003
Ni	58,60	0.0008
Zn	68	0.0162
As	75	0.0027
Ag	107,109	0.0026
Pb	207	0.0028

Table 2: *Concentrations measured in the same sample following analysis of a Zn sample and a cell cleaning procedure*

Conclusions

The Nu Astrum is a new high performance GD-MS that has been tailored to the exacting requirements of the high purity materials industry. The instrument has extremely low detection limits making it suitable for the analysis of even the most refined metals. We have shown that a short and simple cleaning process of the glow discharge cell is sufficient to reduce the background of even a bulk metal to low μg/Kg levels indicating that memory effects after the clean are negligible. Furthermore, unlike some instruments of this type, the Astrum does not suffer from contamination issues outside of the glow discharge cell meaning that maintenance and ongoing running costs can be kept minimal.