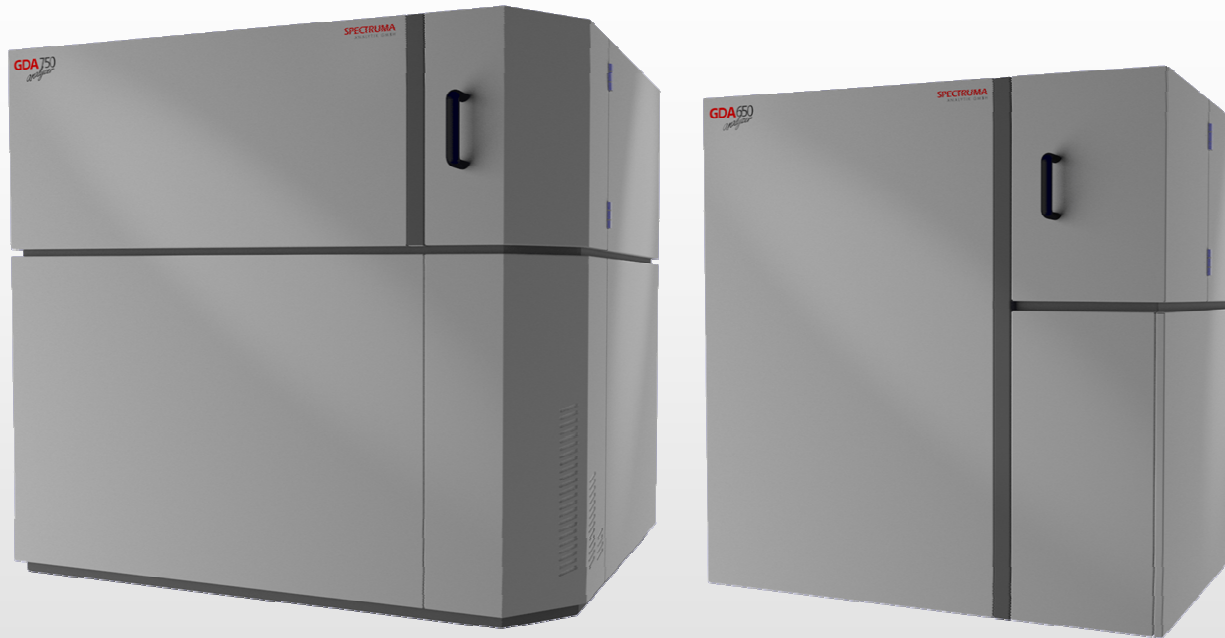


Application note



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Depth profile analysis of non flat/ round samples

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Glow Discharge Optical Emission Spectrometers found their place in the routine analysis of solid materials offering a rapid depth profile analysis on materials with layer structures, e.g. heat treatment, zinc coatings, galvanizing. The ability to perform a proper bulk analysis is not only a prerequisite for that kind of measurement.

There is a wide range of applications of depth profile analysis in production control or incoming inspection. This note shall demonstrate the performance of the Universal Sample Unit while mounted on a GDA in the multi element depth profile analysis of various shaped samples.

This application is optional for all of our products, PMT instruments as well as the CCD spectrometer. Also upgrading of existing systems is always possible if necessary.

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Introduction

The glow discharge source is constructed by the so called Grimm type design with the sample acting as electrode and sealing the source. The instrument can be equipped with anodes of 2.5 mm, 4 mm, and 8 mm inner diameter defining the size of sputter crater and the required sample surface. But this note shall demonstrate the performance of the GDAs in the multi element depth profile analysis of non flat or rounded samples.



Standard USU for round samples DC source



USU for wires < 2.5 mm



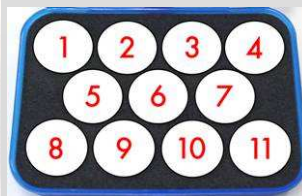
Standard USU for round samples RF source

The analysis of non flat/rounded samples or wires will be done with adaptors.



Description of the shown adaptors.

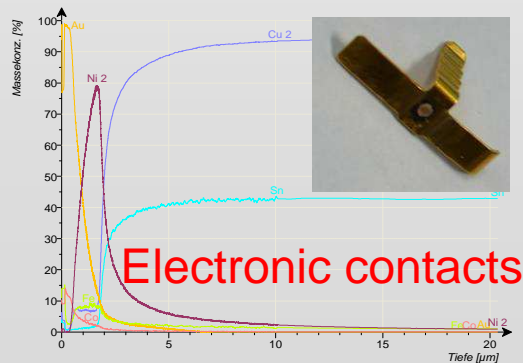
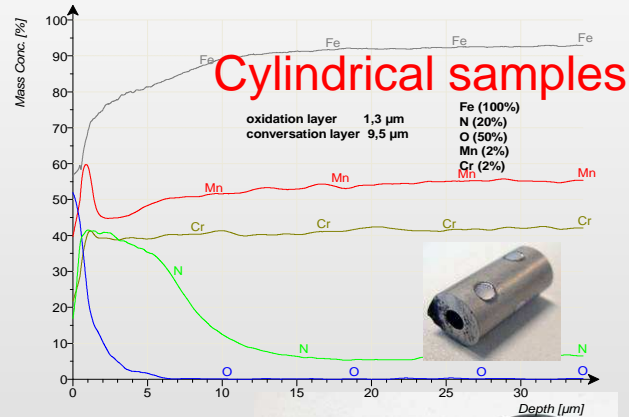
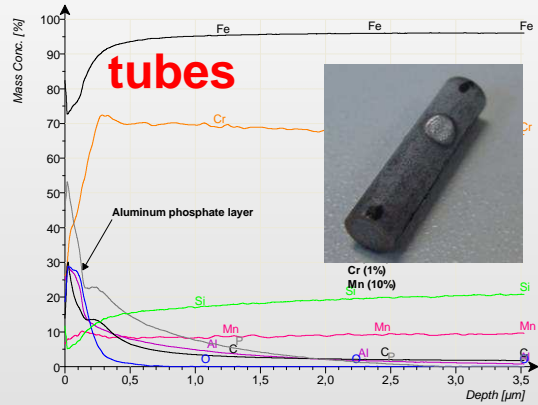
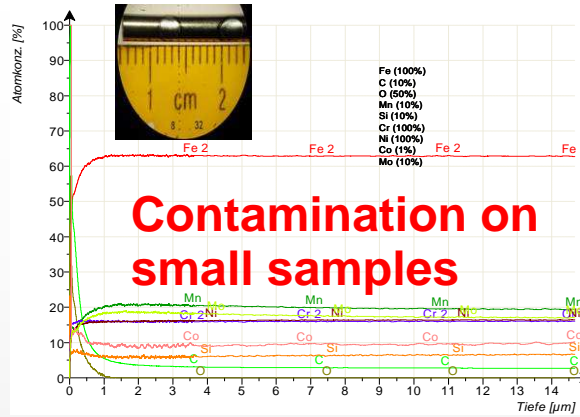
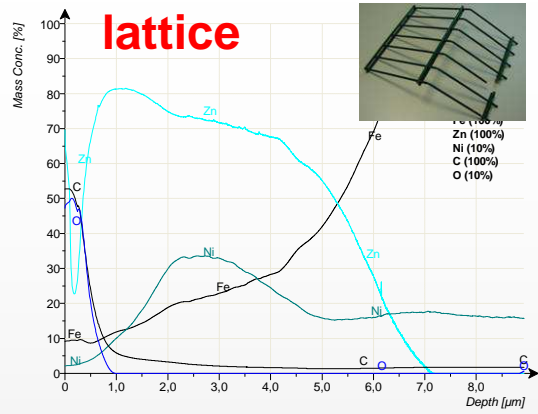
- No.1: for flat samples or recalibration
- No.2-7: various sizes for cylindrical samples
- No.8: for grommets
- No.9-11: balls for various sizes (e.g. of bearings)



Also special geometries are possible

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Introduction



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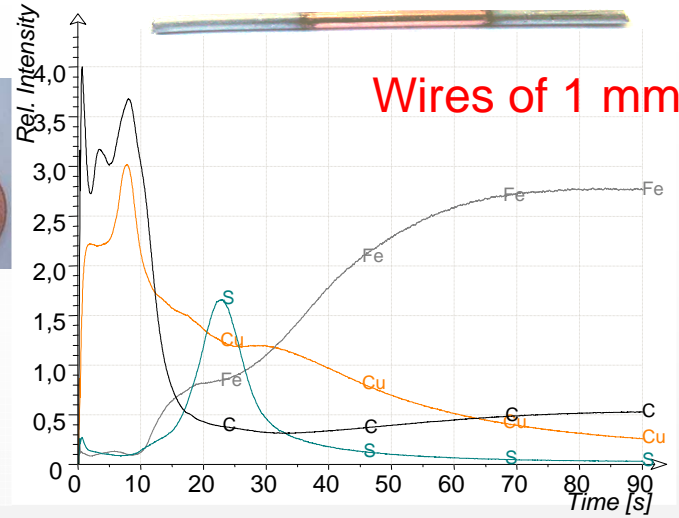
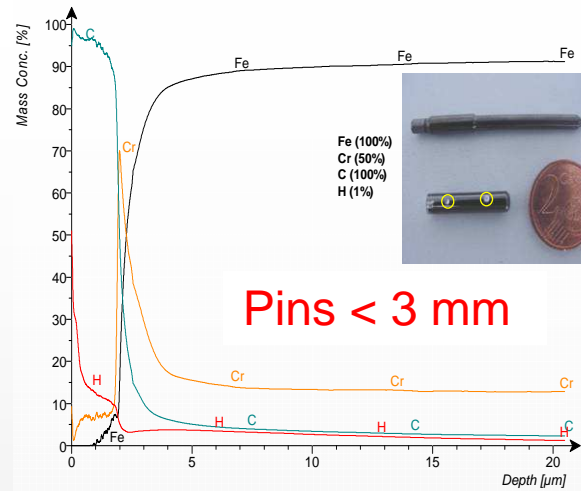
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Broad application range



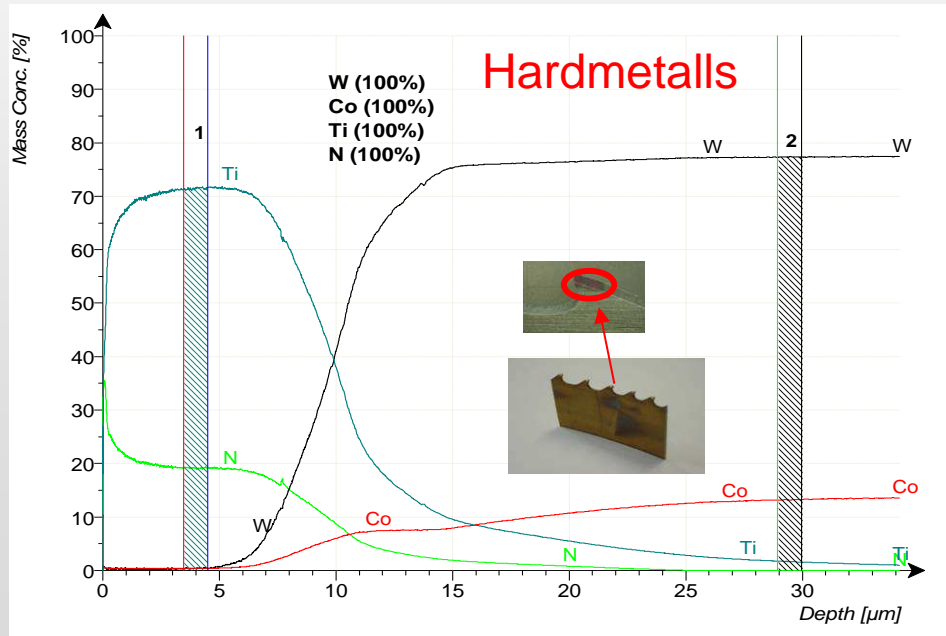
Probe:	10mm								
	Fe [%]	C [%]	Mn [%]	Si [%]	P [%]	S [%]	Cr [%]	Ni [%]	Cu [%]
	Mittelwert	96,63	0,993	0,353	0,221	< 0,001	0,004	1,599	0,032
Probe:	20mm								
	Fe [%]	C [%]	Mn [%]	Si [%]	P [%]	S [%]	Cr [%]	Ni [%]	Cu [%]
	Mittelwert	96,98	0,968	0,276	0,272	0,001	0,009	1,363	0,027

Bulk also possible



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Broad application range



For samples smaller than 2.7 mm diameter the 1mm anode will be used. Here a 1 mm crater will be produced.

Calibration

The GDA650 is factory calibrated using certified reference materials according to the customer's analytical program. One of the big advantages of GD OES is the linearity of these calibrations. Spectruma's HDS mode for the CCD Chips allows the detection of wide concentration ranges using always the best signal evaluation possible and the best line also.

Re-calibration

Setting up samples for drift correction or the adoption to a different set of discharge conditions are provided for each analytical method ordered with the instrument. A recalibration history offers an easy overview of the conditions of the instrument. Furthermore it is possible to include control chart samples in the methods to assure the permanent quality of the analysis.

Sample Preparation

The standard sources require a flat sample surface from 20 mm diameter (8 mm anode) to 6 mm (2.5 mm anode). This surface has to seal the glow discharge source. The quality of this sealing is determining the analytical results especially on those elements that are influenced by atmospheric contamination like oxygen and nitrogen. An universal sample unit for non flat samples is available for each instrument of Spectruma.

The sample surface must be dry and free of oil; even low oil contamination will be detected. For bulk analysis a possibly contaminated surface can be removed by grinding. This should be a dry grinding process using aluminum oxide paper of grain 1200.

Measurement

The discharge conditions for the various applications will be predefined in the analytical methods. Each bulk method will include optimized evacuation and purging time for the source, a preglow time to remove a possibly contaminated sample surface and the analysis time, where the emission line intensity for the elements of interest is evaluated. Depending on the analytical task the total time for one run is ranging from 35 s to 80 s.

Result

Results are presented as a bulk table. They can be stored in an internal database for statistics and printed out using a customer defined layout. A material quality data base is included in the software, the direct comparison with a given quality or the search for matching data is possible directly after the measurement. Data export to standard office applications or LIMB systems is done easily.

Data of depth profiling analysis can be handled in our user friendly software or exported to ASCII files.

Software

The GDA software allows for the creation of so called "jobs". The jobs are actually batches of operations or groups of software instructions linked together which may be user defined or pre-established from the factory. Several separate tasks are combined into one program icon simplifying routine operation. By employing jobs for a given analytical task (analysing heat treated steel sheet for example), even a non-skilled operator can run the instrument and perform all the necessary operations. This can be summarised by placing the sample on the discharge chamber, starting the analysis procedure, waiting for the analysis to be finished, printed and analytical results collected, all with one or two mouse clicks.

Analytical software features such as coating weight, coating thickness, integrated or averaged concentrations for selected elements, can be calculated automatically.

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Summary

The most important and current topics can
be found on our website.

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