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# Depth profile analysis of curved samples using the Universal Sample Unit (USU)

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## GDOES definition and simplified principle

Glow Discharge Optical Emission Spectroscopy (GDOES) is a spectroscopic method for the qualitative and quantitative analysis of metallic and non-metallic solid materials. GDOES allows for the investigation of the elemental composition as well as the layer thickness and layer structure of a sample. Moreover, coating weights can be determined.

The sample is placed into the Glow Discharge Source and switched as cathode. The Glow Discharge Source is filled with argon gas under low pressure. Direct Voltage is applied between a hollow anode and the cathode (≙ sample). Due to the energy input of the DC voltage the argon atoms are ionized resulting in the formation of a plasma. Argon cations are accelerated towards the negative sample surface and knock out some sample atoms (“sputtering”).

The knocked out sample atoms diffuse into the plasma where they collide with high-energy electrons. During these collisions energy is transferred to the sample atoms promoting them to excited energy states (Figure 1). Returning to the ground state the atoms emit light with a characteristic wavelength spectrum.
In the spectrometer the light is dispersed into its spectral components, which are registered by the detection system. The intensity of the lines is proportional to the concentration of the corresponding element in the plasma.

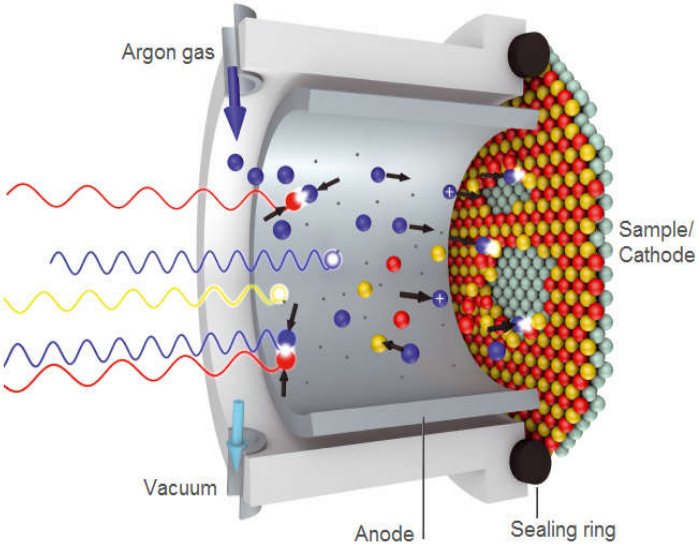


Figure 1. Scheme of the glow discharge excitation process

## Introduction

As mentioned before, Glow Discharge Optical Emission Spectrometry (GDOES) is a well-established method for the investigation of solid materials.

Usually, the GDOES analysis requires a flat sample surface. But a lot of samples do not display a standard geometry with a flat surface. To address this problem, Spectruma Analytik developed the so-called Universal sample Unit (USU). The USU is optionally available.

This note demonstrates the performance of the GDAs in the multi element depth profile analysis of variously shaped samples.

## Why USU?

The Glow Discharge Source (Figure 2) is a Grimm type discharge tube characterized by a special arrangement of the electrodes: The two electrodes of the DC Current Source are set up of a cylindrical hollow anode and the sample as cathode. The sample must seal the anode tightly so a vacuum can be generated. Thus, a flat and preferably smooth sample surface is required.

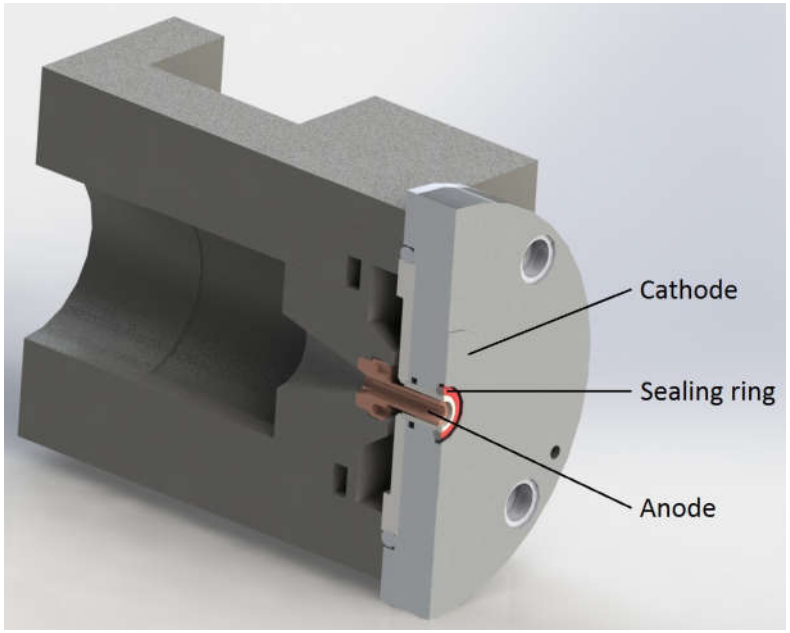


Figure 2. Cross section of a Glow Discharge Source

The Glow Discharge Analyzers (GDA) can be equipped with anodes having inner diameters of 2.5 mm, 4 mm or 8 mm. The inner diameter defines the size of the measuring spot (“sputter crater”).
The diameter of the sample must be at least few millimeters larger than the diameter of the sealing ring of the corresponding anode. For instance, using the 2.5-mm-anode requires a sample surface area of at least 6 mm.

**Smaller samples and curved samples can be analyzed by the use of the Universal Sample Unit, only.**

## Finding the right adaptor

Spectruma Analytik offers several different adaptors for various forms like cylinders and spheres of different sizes. But also customized adaptors can be produced upon request.
The sample is placed into the fitting adaptor (Figure 3). The generation of a vacuum is provided by the use of special plastic caps (Figures 4 and 5). Moreover, there is a Universal Sample Unit specially for wires (Figure 6).


Figure 3. Various Adaptors:

| | |
|---|---|
| No. 1: | for flat samples |
| No. 2 - 7: | for cylindrical samples of different sizes |
| No. 8 -11: | for spheres of different sizes |
| No. 12: | customized construction for a special geometry |

Figure 4. Standard USU with cap; DC source

Figure 5. Standard USU with cap; RF source

Figure 6. USU for wires < 2,5 mm

## Applications

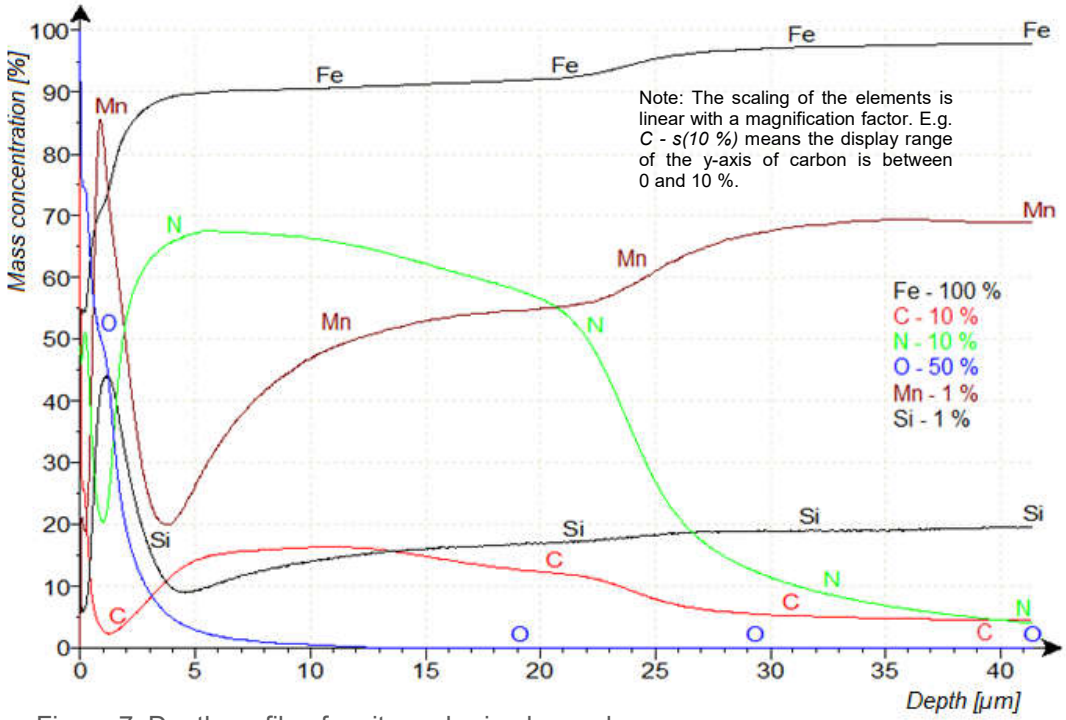


Figure 7. Depth profile of a nitrocarburized sample

Figure 8. Curved nitrocarburized sample with analysis spots beside a European 2-cent coin (18.75 mm diameter) for size comparison

Figure 7 shows the depth profile of a curved nitrocarburized sample (Figure 8) with an oxide layer on the surface. The typical courses of the elements nitrogen and carbon can easily be monitored using GDOES.

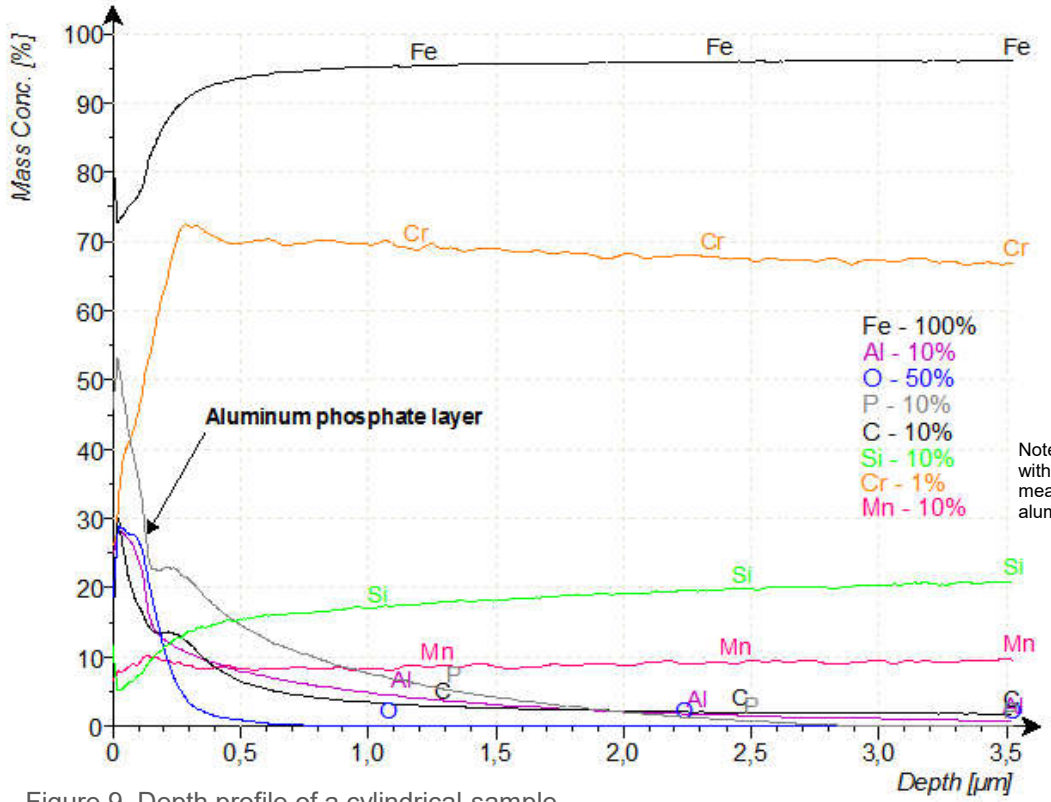


Figure 9. Depth profile of a cylindrical sample

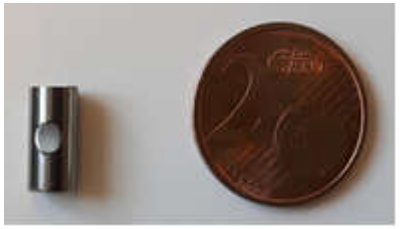

Figure 10. Cylindrical sample with crater

Note: The scaling of the elements is linear with a magnification factor. E.g. *Al - s(10 %)* means the display range of the y-axis of aluminum is between 0 and 10 %.

Figure 9 depicts the depth profile of a cylindrical sample (Figure 10) with an aluminum phosphate layer on the surface. Among other elements, traces of chromium were detected in the bulk material.

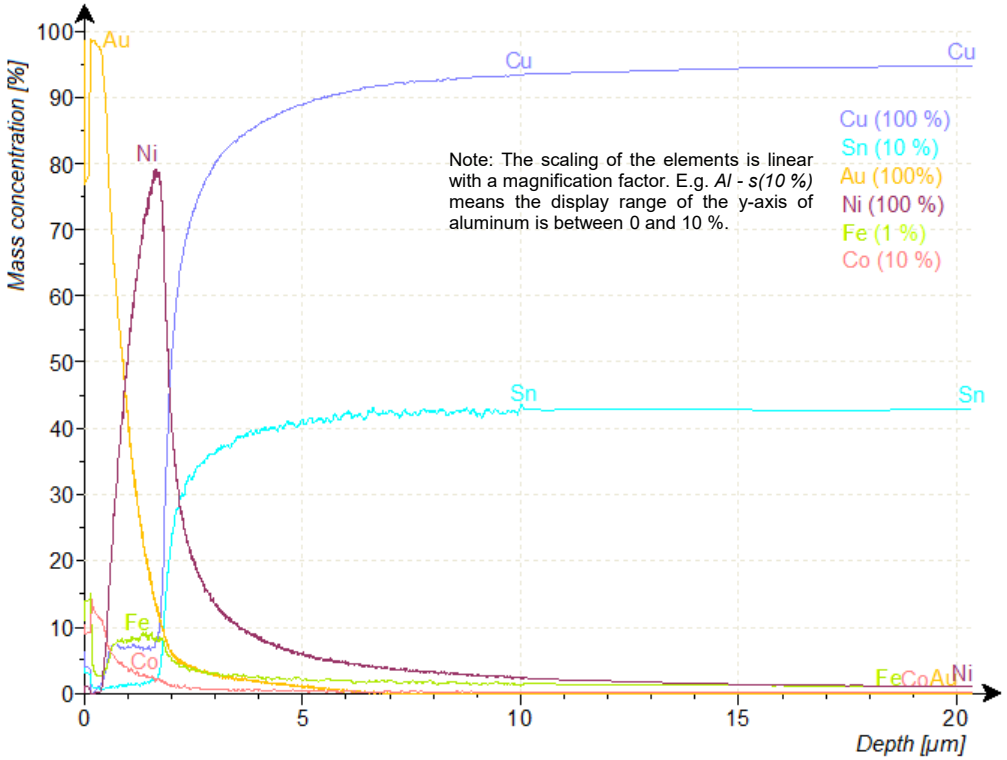


Figure 11. Depth profile of an electrical contact

Figure 12. Electrical contact with crater

Figure 11 shows the depth profile of an electrical contact (Figure 12). There is a gold layer of approximately 1 µm thickness and a nickel layer of about 1.2 µm thickness on the surface.

## Analysis with the 1 mm anode

Small samples can be analyzed by the 1 mm anode. The 1 mm anode can only be used in combination with the Universal Sample Unit. Figures 13 und 14 show examples for small and uneven components, which were successfully analyzed by the 1 mm anode. The measuring spots (craters) are highlighted by red arrows.

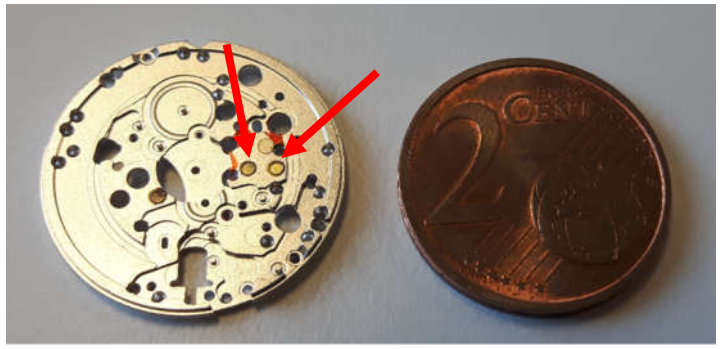

Figure 13. Uneven component with a 1 mm crater (marked by red arrow) beside a European 2-Cent-Stück (diameter: 18.75 mm) for size comparison

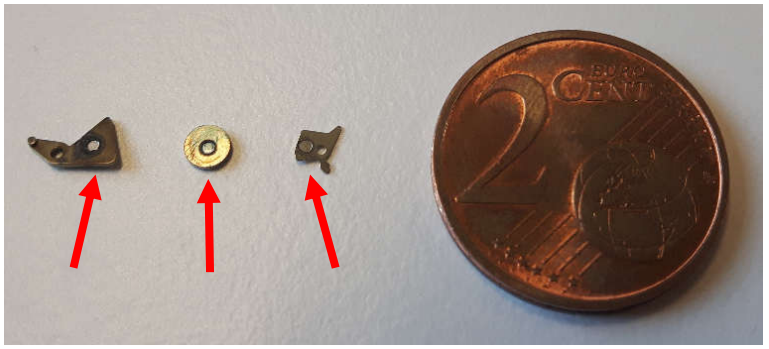

Figure 14. Tiny components with 1 mm craters (marked by red arrow) beside a European 2-Cent-Stück (diameter: 18.75 mm) for size comparison

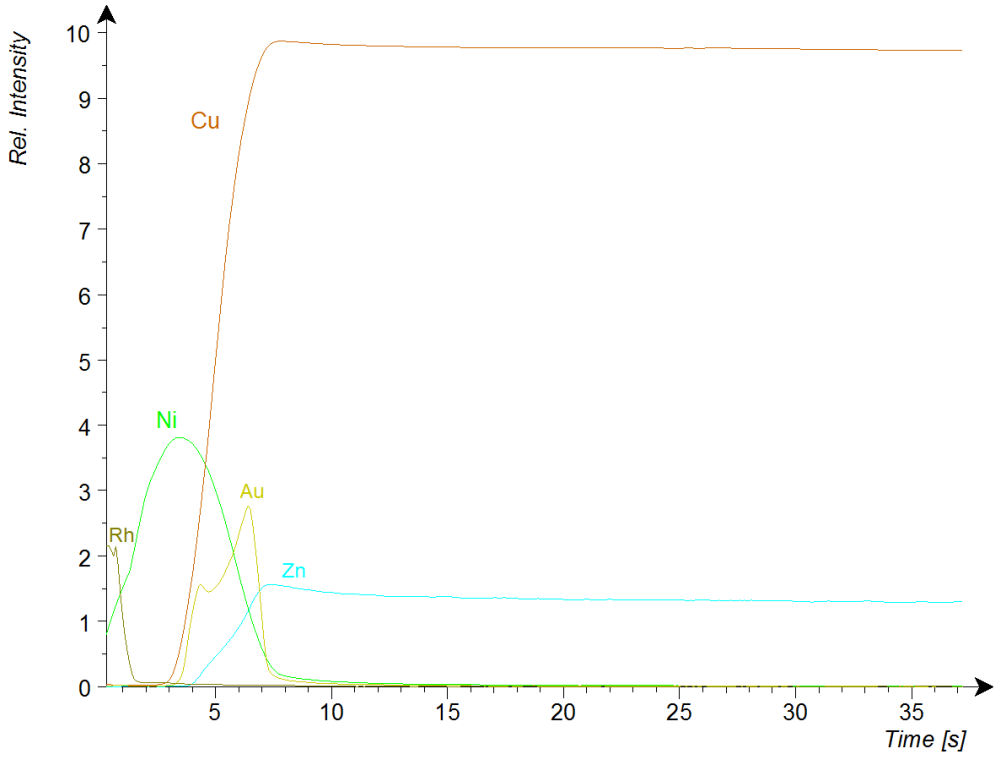


Figure 15. Depth profile of a small, uneven component

Figure 16. Component from Figure 13 zoomed by a microscope.

Figure 15 shows the depth profile of the component from Figures 13 und 16 with the relative intensity plotted versus the analysis time. The layers consisting of rhodium, nickel and gold are highly resolved.

## Analysis of wires

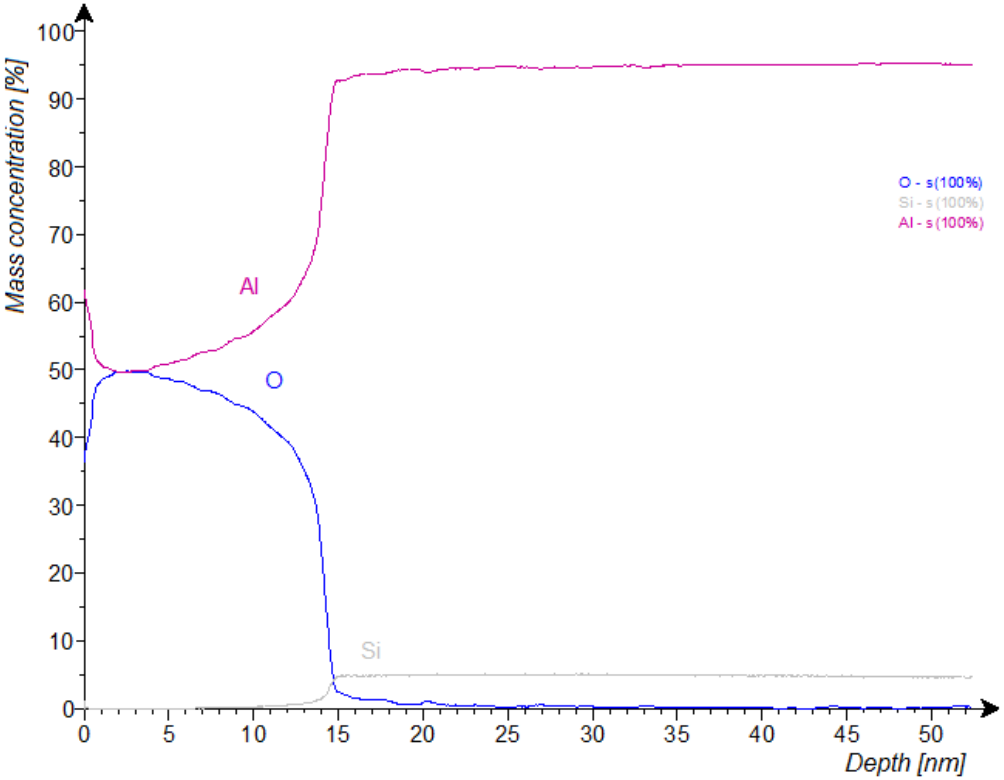


Figure 17. Depth profile of a wire

In the diagram, an aluminum oxide layer of about 13 nm thickness is clearly discernible on the surface. The wire contains 5 % silicon.

Figure 17 displays the results of the analysis of an aluminum wire of 1 mm diameter. To analyze samples of such sizes and geometries the Universal Sample Unit for wires is used (Figures 6 and 18).

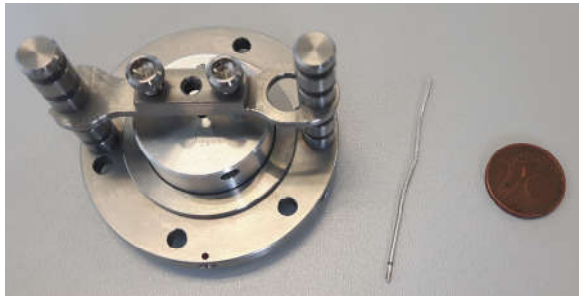

Figure 18. Aluminum wire beside the Universal Sample Unit for wires and a 2-cent-coin for a size comparison

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## Summary

For standard measurements with the Glow Discharge Analyzers by Spectruma Analytik the samples must exhibit a flat surface and have a certain minimum size so they can seal the anode and a vacuum can be created.
Samples not fulfilling these criteria can be analyzed by the so-called **Universal Sample Unit**. Uncommon sample shapes and/or their small sizes are compensated by placing them into fitting adaptors. The use of special plastic caps enables the generation of a vacuum. Also wires can be analyzed using a special sample unit.

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