Spectroscopy Performance Note

Quantitative Depth Profile (QDP) Analysis of Plated Samples

- **Plating depths and coating weights**
- **Composition of the coating and substrate**
- **Surface treatments**

Improved corrosion resistance, increased wear resistance, or a decorative finish can be achieved by plating of one or more metallic layers onto a substrate. The most common method of plating is by electrodeposition in which the plating occurs by the action of an electric current. A negative charge is applied to the object to be coated and it is immersed into a solution which contains a salt of the metal to be deposited. The positively charged metallic ions of the salt are attracted to the object and are reduced to their metallic form on the surface, creating the plated metal layer. A number of metals such as Cu, Cr, Ni, Cd, Ag, Sn, and Au can be applied by carefully controlled plating baths to yield layers of the desired thickness and properties. Another method of deposition is electroless nickel plating where the plating reaction occurs by an autocatalytic chemical reaction.

Tinplate

Tin electrodeposits provide ductility, solderability, and corrosion or tarnish resistance to the surface of a number of alloys. For copper and nickel substrates they provide sacrificial protection. A common application is tinplated steel for the manufacturer of food cans. The plot shows three replicate analyses of tinplated steel. The table provides the Sn depth in micrometers (μm) and the coating weight in grams per square meter (g/m²). Each analysis is completed in less than 60 seconds. Tinplate coatings are fairly thin and are usually under 10 g/m² in coating weight.
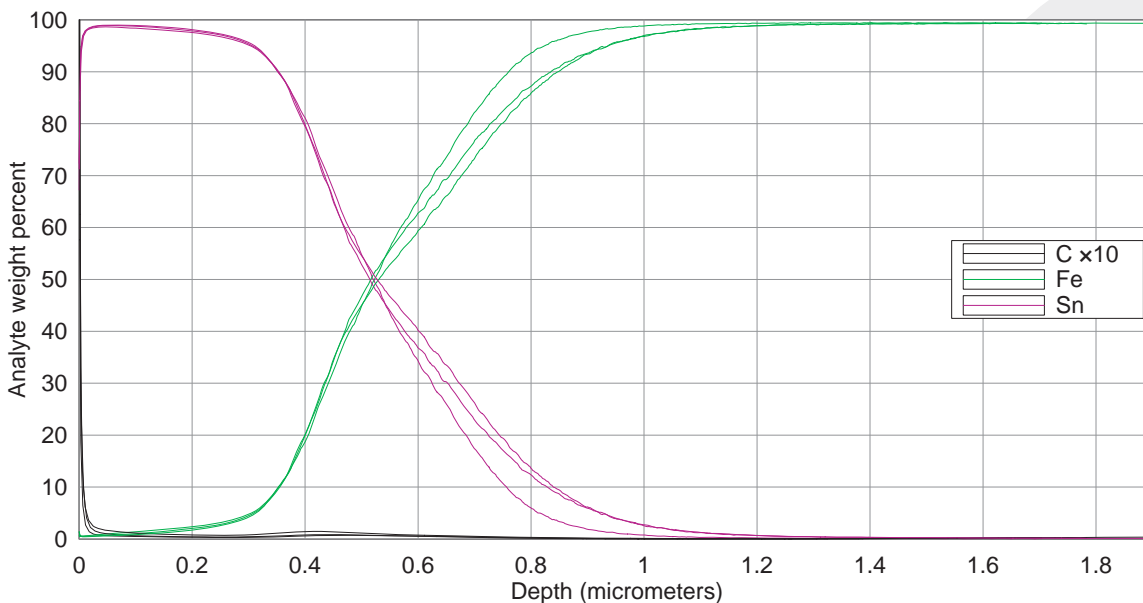
| Notes	Sn Depth, µm	Sn Coat Wt, g/m²
Tinplate-1	0.52	3.95
Tinplate-2	0.51	4.11
Tinplate-3	0.52	4.18
Mean	0.52	4.08
Rsd	1.12	2.89

GDS-Series

Chromium, Nickel and Copper Plating

Electrodeposited metals can be applied singly or in multiple layers. Depending on the application the layers may provide corrosion resistance, hardness, adhesion, or decoration. The next plots show decorative chromium plating with nickel and copper under-layers on a zinc alloy substrate. The decorative chromium layer is very thin (<1 μm), as seen in the first plot.
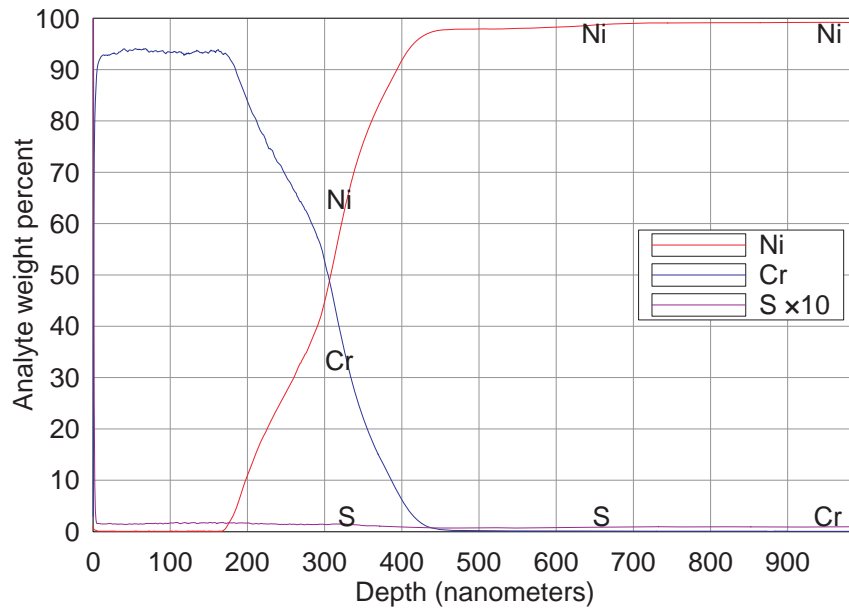

The second plot shows the remainder of the coating layers and finally the zinc substrate. Two different nickel layers are shown—the first layer contains sulfur. The table gives the thickness of each layer. Detailed analysis to a depth over 100 μm is collected in under 10 minutes.
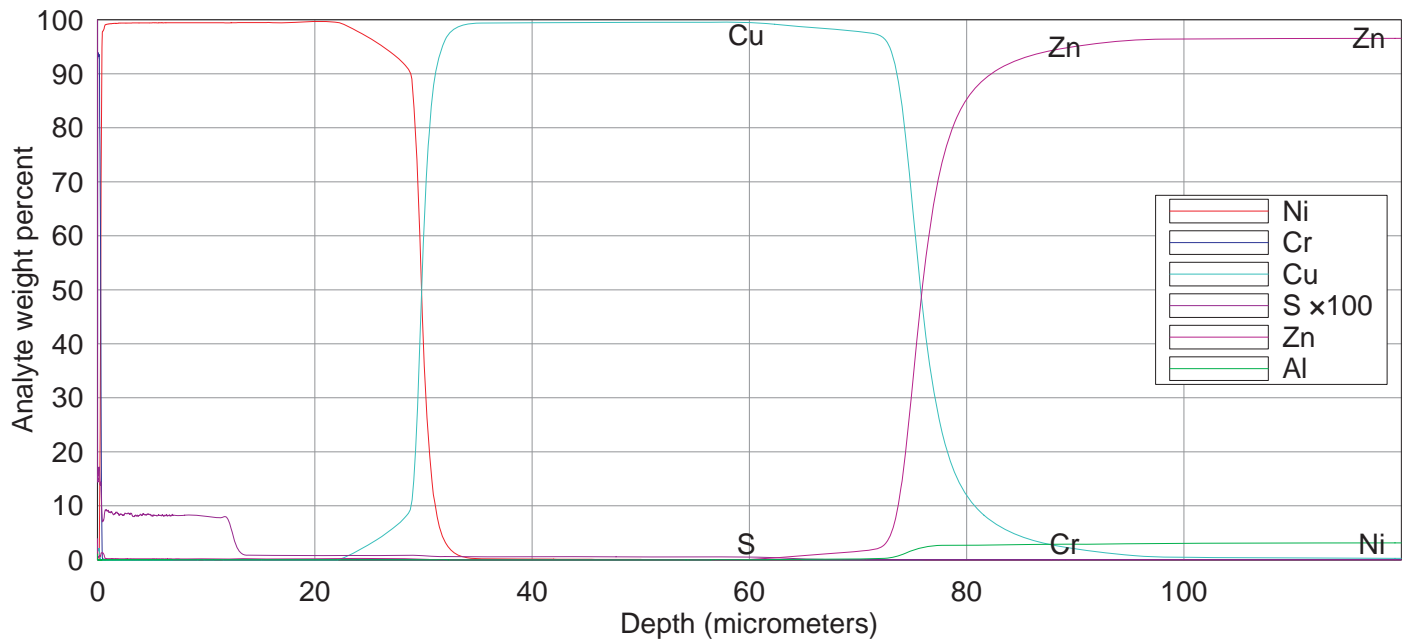
| Name	Cr Depth, nm	Ni-1 Thickness, µm	Ni-2 Thickness, µm	Cu Thickness, µm
Multilayer	308	12.17	17.62	45.94

Electroless Nickel Plating

Electroless nickel is an engineering coating comprised of nickel and phosphorus which provides good corrosion and wear resistance, uniformity, and low labor costs. The limitations include high chemical cost, brittleness, and slower deposition rate, when compared to electroplating. The following plots show an electroless nickel coating on an aluminum alloy substrate for a hard disc. The first plot shows the full depth of all the coating layers into the substrate. The second plot shows the surface layers which include C, O, Co, Cr, and Pt.
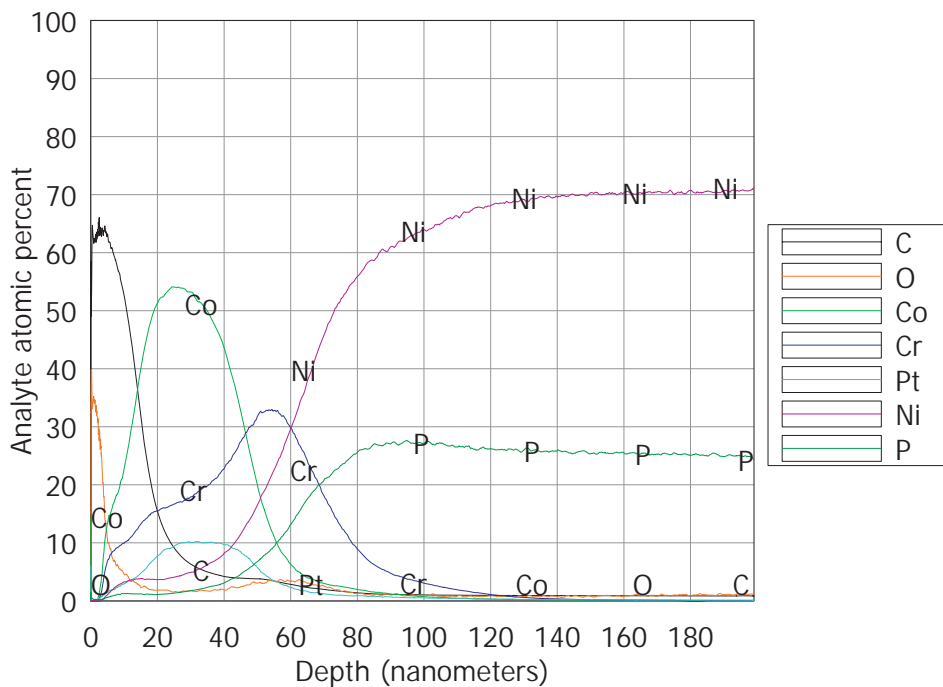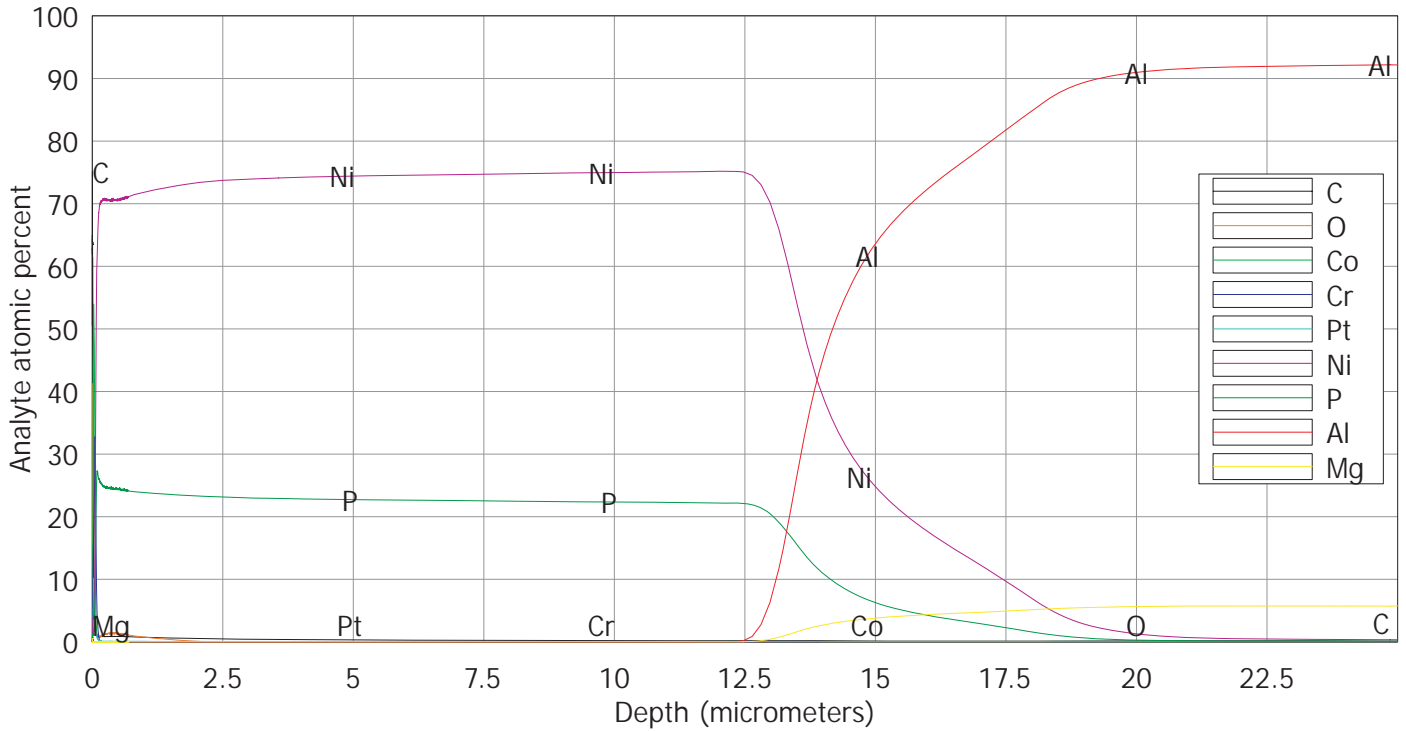
Gold Plating

Electrodeposited gold is in high demand in the electronics-related industries. In addition to wear and corrosion resistance, Au-plated surfaces also offer low electrical contact resistance, good solderability, weldability, and high infrared emissivity. The following plot shows an Au-plated layer over nickel and copper under-layers. The substrate is a circuit board which is nonconductive; however, analysis can be performed with a DC lamp well into the copper under-layer. The table provides the depths of the Au and Ni coatings.
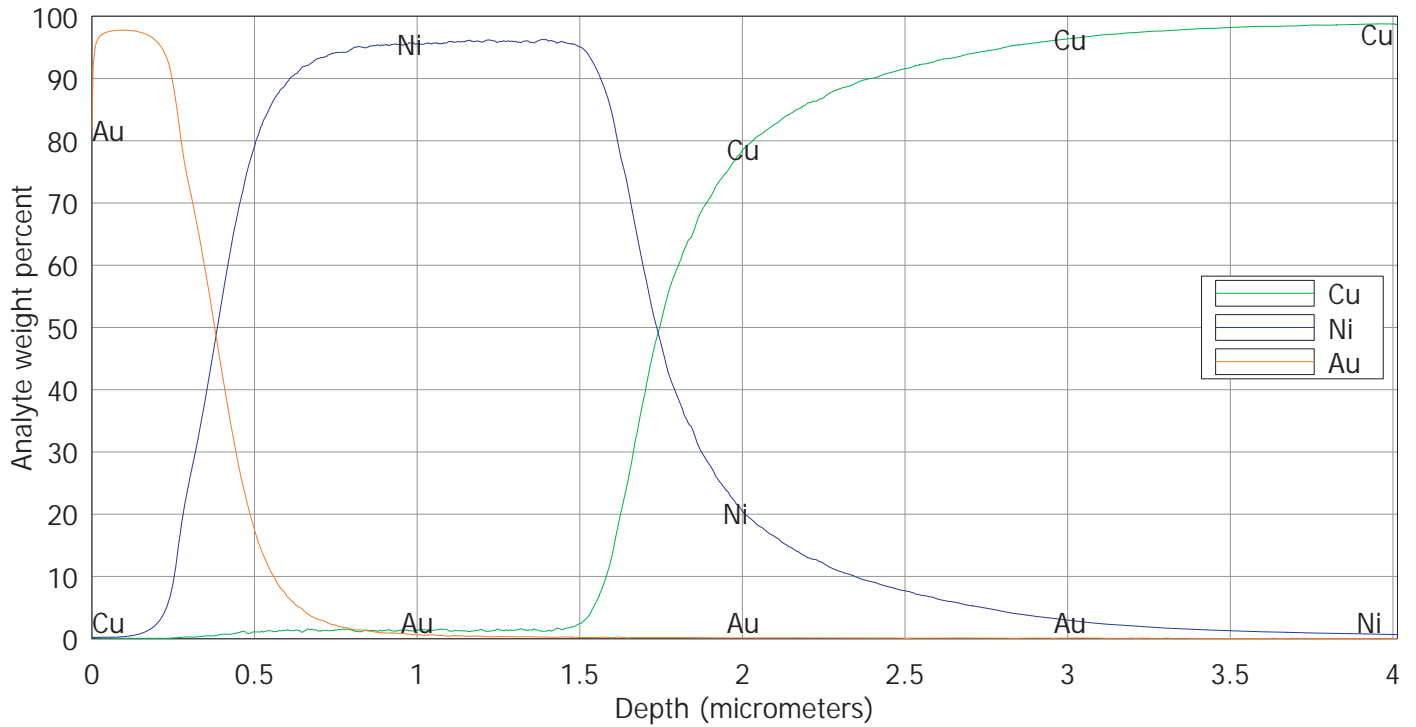

Notes	Au depth, μm	Au coat wt, g/m²	Ni Depth, μm
Board	0.38	5.80	1.36

Understanding the Glow Discharge Source

The Sputtering Process

- The Glow Discharge Spectrometer (GDS) lamp provides a low pressure argon environment (typically 5-10 Torr) over the sample surface
- A high negative potential (typically -800 to -1200V) is applied to the sample. The sample thus becomes the cathode
- Spontaneously produced Argon ions (Ar^+) are accelerated across the anode/cathode gap by this potential
- The collision of Ar^+ ions with argon gas molecules causes plasma formation and further production of Ar^+ ions. This plasma is called a glow discharge
- Some of these high velocity Ar^+ ions reach the sample surface where they sputter (or mill out) materials uniformly from the sample substrate
- Some of this sputtered material diffuses into the glow discharge plasma where it is dissociated into atomic particles and finally excited
- The light emitted from these excited state species as they collapse back to a lower energy level is characteristic of the elements composing the sample
- The wavelengths and intensity of the light emission are used to identify and quantify the composition of the sample
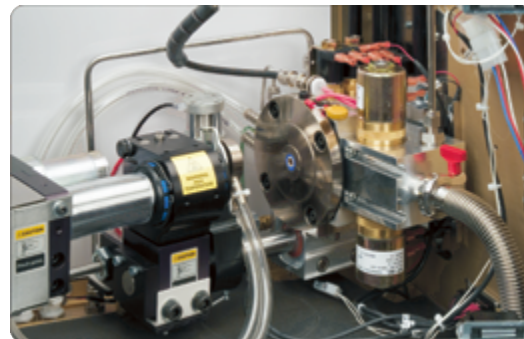
GDS Advantages

- Layer-by-layer removal of material allows for qualitative and quantitative depth profile analysis
- Separation of sampling (sputtering) and excitation resulting in:
  - Freedom from metallurgical history
  - Fewer matrix effects
- Grimm-type Lamp design provides lowered self-absorption and material re-deposition
- Linear calibration curves with wide dynamic range
- Fewer lines required to analyze full concentration range
- Linear calibrations require fewer standards for calibration
- Fewer spectral interferences due to:
  - Narrow emission lines
  - Excitation of almost exclusively atom lines
- Very little sample-to-sample carry-over allows quick matrix changes
  - Automatic cleaning between samples
  - No sputtering of anode or other lamp components
- Low reference material consumption
  - More burns before required resurfacing
  - Shallower burn spots requiring less material removal during resurfacing
- Low gas and other consumable consumption
- Very easy to operate
- Quiet, clean, and low maintenance
- Small footprint—fits through standard lab door