Spectroscopy Application Note

QDP Quantitative Depth Profile Analysis
Hot Dip Galvanization

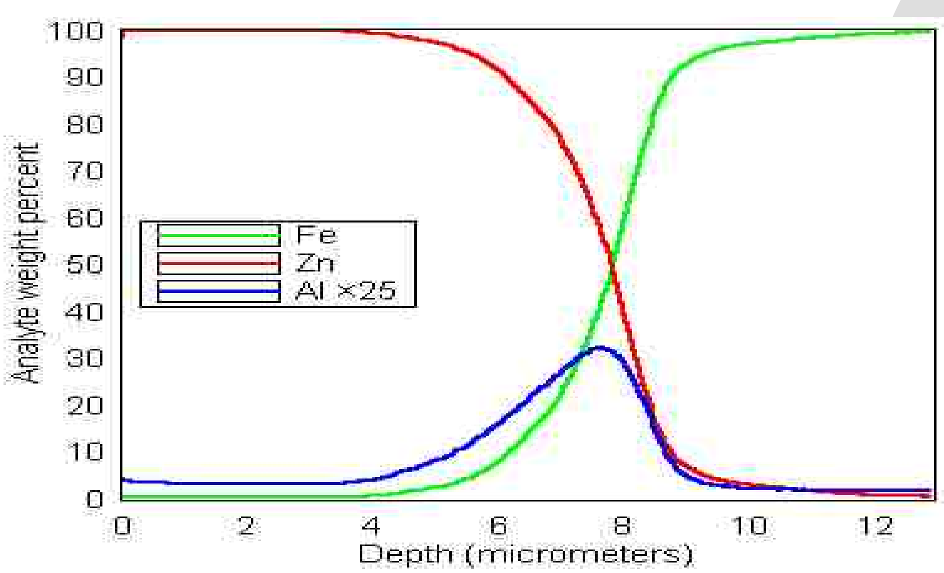
- **Zinc Thickness and Coating Weight Determination**
- **Total Aluminum Determination**
- **Surface Aluminum Determination**

GDS-400A

Hot Dip Galvanization is a well-proven technology used to protect steel sheets against corrosion. The steel sheet is coated by immersion in a molten zinc bath. It is a hot process which involves chemical reactions at the zinc/steel interface. The presence of aluminum in the zinc is characteristic of the Hot Dip Galvanization. The thickness of the zinc coating, the coating weight and the measurement of the total and surface aluminum are major tests made to check the quality of Hot Dip Galvanization. These tests are presently made by chemical techniques and are time consuming. The glow discharge-optical emission spectroscopy (GD-OES) technique can verify the quality of such products in a short period of time and solve some problems occurring during the process.

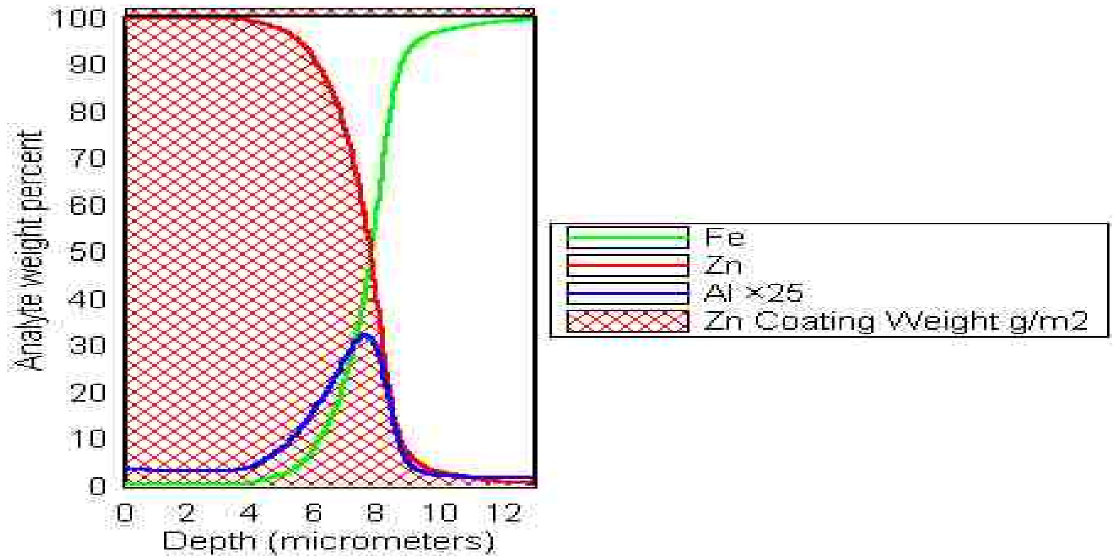
GD-OES Saves Time and Money for Hot Dip Galvanization Quality Control

Zinc Thickness and Coating Weight Determination: Depth Profile Analysis will provide the continuous chemical composition of the materials from the surface to substrate. A diagram representing the concentration on the Y-axis versus the depth on the X-axis obtained after calibration is shown below.
This is an example of an 8 microns thick, Hot Dip Galvanization. The complete profile from the surface to the substrate (approximately 13 microns) was obtained in less than one minute. The time of analysis depends on the thickness of the coating. For a thicker galvanization, such as 20/30 microns, the analysis time will be around two to three minutes. The software automatically calculates the Zn thickness and the Zn coating weight.



GDS-Series

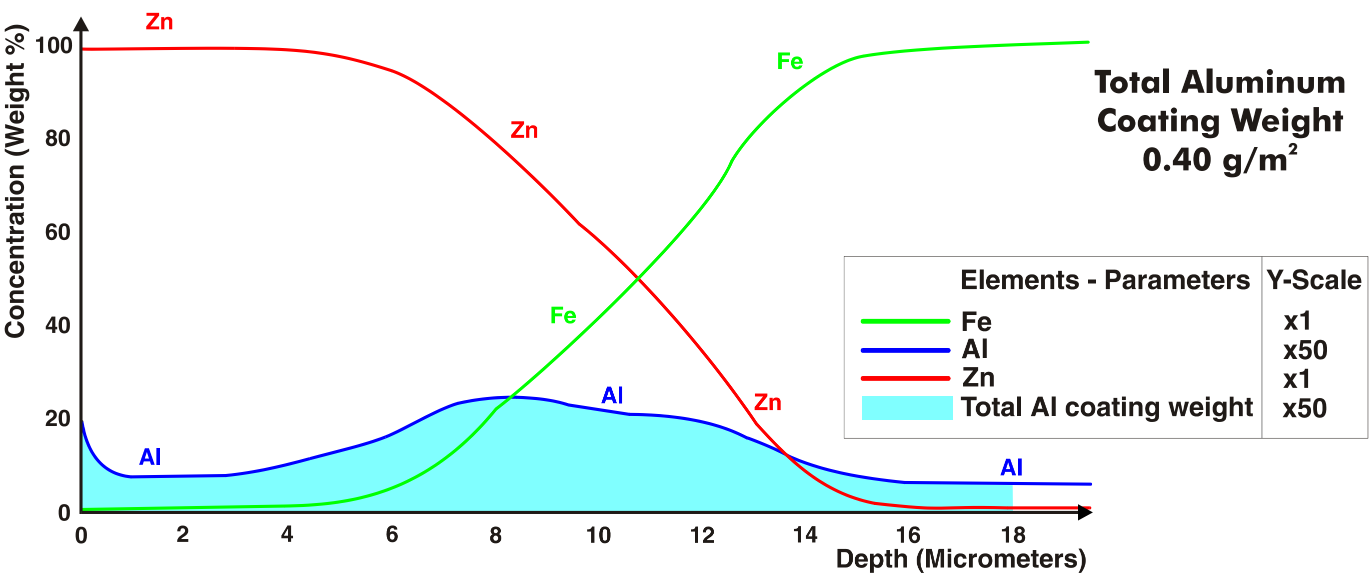
An example of the integration area for the coating weight determination and the result of the calculation follows.



Name	Zn Thickness micron	Zn Coating Weight g/m2
Hot Dip Galvanization	7.8713	54.828

The Zn thickness and the coating weight are calculated instantaneously after the acquisition of the profiles. The flexibility of the software allows the calculation of many other parameters which can be of interest to the operator, such as the average chemical composition of the coating and the substrate.

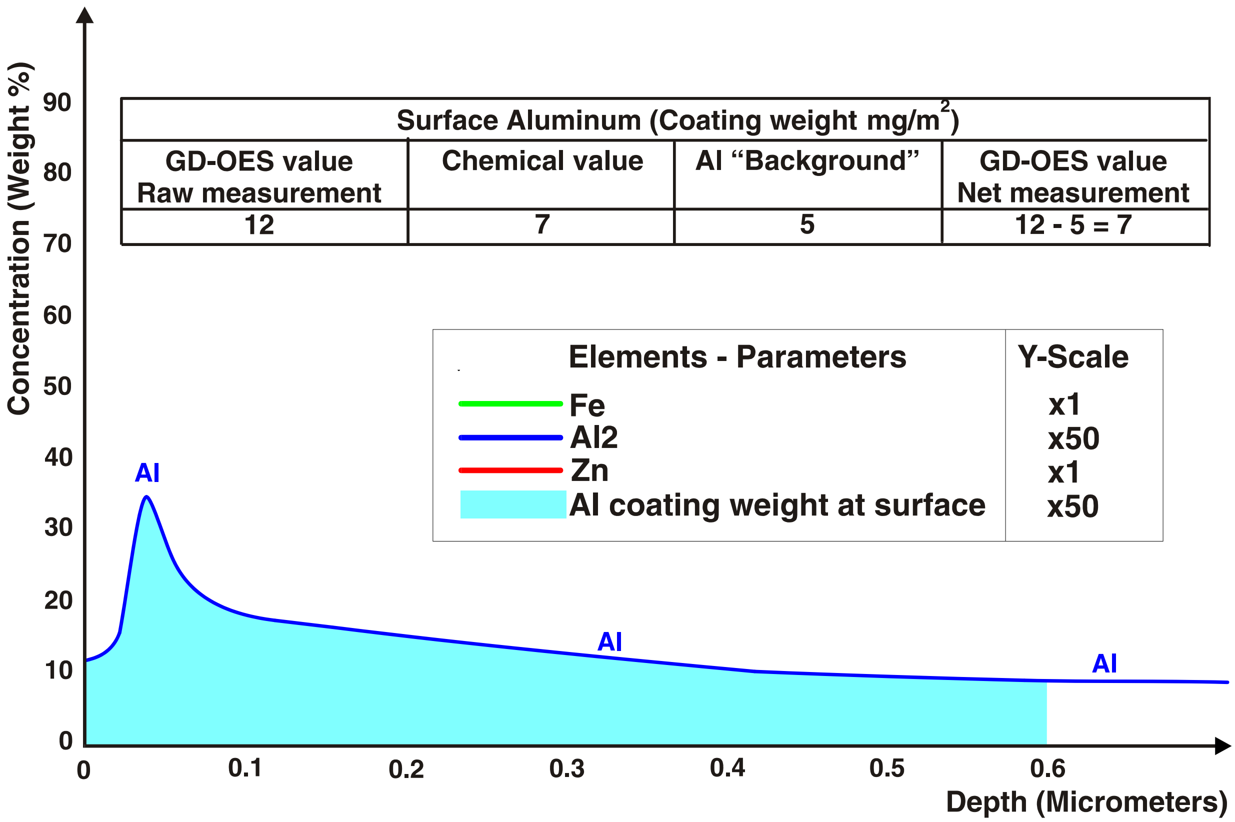
Total Aluminum Determination: As discussed previously, the total aluminum determination is a very important parameter. After the acquisition, the software will instantaneously calculate the total aluminum coating weight. The following is an example of an 11 microns thick, Hot Dip Galvanization. The shaded area is defined for the total aluminum coating weight determination.



The total Aluminum determination (made by chemical method) was 0.38 g/m^2. The result obtained from the depth profile analysis is 0.40 g/m^2. The correlation between the two techniques is excellent.

Surface Aluminum Determination: The surface of hot dip galvanized steels is very complex and depends largely upon the composition of the galvanization bath and surface treatments such as degreasing and chromate conversion. Some investigations have shown there is a relationship between the amount of aluminum at the surface of the galvanization and the strength of the epoxy-galvanized steel bond. Therefore, a systematic control of the aluminum content at the surface of hot dip galvanized steel sheets becomes necessary before applying any additional coating. Below is a zoom of the previous diagram showing the aluminum peak at the surface of the galvanized section.

Surface Aluminum (Coating weight mg/m^2)			
GD-OES value Raw measurement	Chemical value	Al "Background"	GD-OES value Net measurement
12	7	5	12 - 5 = 7



The calculation of the surface aluminum coating weight obtained by integration of the shaded part indicates 12 mg/m^2. However, it is necessary to take into account the value of the "background" due to the normal concentration of metallic aluminum in the coating (5 mg/m^2). After this correction, the result given by GD-OES is 12-5 = 7 mg/m^2, which is exactly the value obtained by chemical techniques.

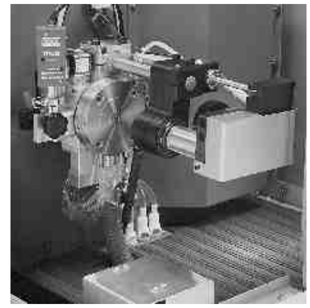
Chemical methods used to quantify the surface aluminum are based on the difference of solubility between aluminum (oxidized) in the surface and metallic aluminum in the coating. These methods are difficult to employ by current quality control practices.

Application Summary: The ability to quickly provide zinc thickness/coating weight and total/surface aluminum content, makes GD-OES an ideal means of systematically evaluating galvanized steel production. Using GD-OES has proven to save time and money for quality control.

Understanding the Glow Discharge Source

The Sputtering Process

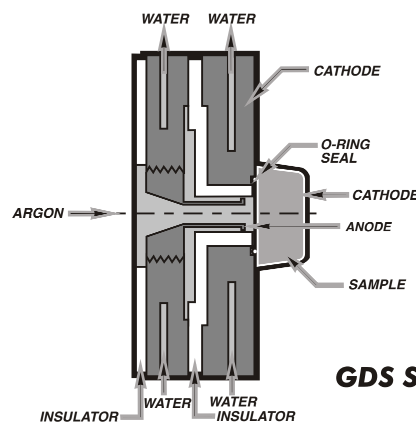
- The Glow Discharge Spectrometer (GDS) lamp provides a low pressure argon environment (typically 5-10 Torr) over the sample surface.
- A high negative potential (typically -800 to -1200V) is applied to the sample. The sample thus becomes the cathode.
- Spontaneously produced Argon ions (Ar^+) are accelerated 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 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 Lamp

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
- Very linear working curves
- Fewer lines required to analyze full concentration range
- Linear calibrations require fewer standards for calibration
- Fewer spectral interferences due to:
  - Narrower emission lines because of less Doppler broadening
  - 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



GDS Source

LECO Corporation
3000 Lakeview Ave. • St. Joseph, MI 49085-2396
Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • *ISO-9001 No. FM 24045*

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