
Glow Discharge-Optical Emission Spectrometry (GD-OES) is a fast, easy-to-use analytical procedure which can produce detailed information on both metallic and organic coatings at a lower cost than alternative methods. It is suitable for use in production control environments as well as for research investigations.

For example, the steel industry is required to supply materials with precise surface attributes for corrosion and wear resistance. Many types of surface treatments and surface coatings are presently used in the production processes, and a method for the quality control of the surface is necessary.

GD-OES is an analytical method for the determination of elemental composition in bulk analysis with the capability of surface depth profiling. Atomization is performed by sustaining a glow discharge plasma in argon where the sample is the cathode. The technique can provide continuous qualitative and quantitative profiles from the surface (~10 nm) down to the substrate (tens of µm). Only a few minutes are necessary to obtain the chemical composition of each layer with excellent in-depth resolution (of some nanometers).

This technique provides important information for the development of new coatings and surface treatments. It may also be used in the laboratory for research investigations. The concept of depth profiling using the LECO Surface Analyzer was described by Maul and Weiss, in Asia Steel 1997, pp 251-253, in an article which showed how the technique could be used to determine the parameters of metallic coatings on steel substrates.

Workers at IRSID (part of the French USINOR Group) have shown that depth profiling can provide various types of information including chemical composition of coatings and substrates, coating thickness, coating and layer homogeneity, and element migration at interfaces. In this article we shall present one application, in which emission intensity or concentration is plotted against sputtering time or depth, for the investigation and quality control of steel surfaces and surface treatment processes.

The surface of hot-dipped galvanized steel is a very complex structure, which depends largely upon the composition of the galvanizing bath and on the surface treatment, for example, degreasing and chromate conversion. Various studies have been undertaken with the aim of correlating surface composition with properties. Researchers at IRSID and elsewhere have examined surface reactivity and bonding characteristics in comparison with those of pure zinc and electro-deposited zinc coatings. These studies have demonstrated the major role played by surface contamination in epoxy-zinc coated steel bonds. The presence of aluminum at the surface of the galvanized sheet has a very important influence on the adherence of the epoxy layer deposited on the galvanization. The epoxy-zinc bond is modified depending on the aluminum concentration at the surface.

By using X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared measurements (FTIR), it was possible to demonstrate the detrimental effects of surface aluminum with respect to the adsorption of the hardener (dicyandiamide) present in the epoxy coating. While these methods are highly effective for the study of coatings, they cannot be used for production control. To evaluate the surface aluminum on galvanized steels, GD-OES can provide in-depth elemental profiles in a short analysis time. Thus, a systematic evaluation can be made of the coating process.

A statistical study by SOLLAC (part of the French USINOR Group) indicates that there is a relationship between the amount of aluminum at the surface of the galvanized steel and the strength of the bonded joint between the epoxy and the galvanized steel. In this work the resistance of the joint was measured by single-lap shear test (Abbel et. al, 1996). The results for surface aluminum were qualitative (peak areas) but the correlation with resistance measurements was sufficient to highlight the deleterious effect of aluminum contamination.

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

Recent progress in achieving a more quantitative approach demonstrates that GD-OES can be used instead of the tedious chemical measurements. This represents an important improvement in production quality control.

| Sample | Chemical Value | GD-OES Value |
|---|---|---|
| #1 | 6.3 | 6.6 |
| . | 7.8 | 6.9 |
| . | 7.0 | 7.3 |
| . | 7.7 | 7.1 |
| #1 Mean | 7.2 | 7.0 |
| #2 | 23.3 | 20.8 |
| . | 22.9 | 22.6 |
| . | 22.6 | 20.2 |
| . | 18.1 | 21.5 |
| #2 Mean | 21.7 | 21.3 |
| #3 | 13.2 | 13.2 |
| . | 13.0 | 12.9 |
| . | 12.8 | 12.4 |
| #3 Mean | 13.0 | 12.8 |

Compare results obtained by the chemical and the GDS-EOS method; these data were compiled by Renault in France as part of a collaborative test made by the French GD-EOS User's Group.

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