Bulk Analysis of Hot Dip Galvanizing Bath Using the GDS500A

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Instrument: GDS500A

Introduction

Hot dip galvanization is an important passivation process in which steel is coated with a thin zinc layer thereby protecting it from harmful environments that lead to corrosion. Control of the hot dip bath chemistry is crucial to obtain the desired corrosion resistance properties as well as surface finish. For example, the crystalline surface finish, or spangle, was formerly controlled by the addition of lead in the bath which promoted a more fluid melt and more aggressive spangle formation. Additionally, current environmental recommendations are to keep the bath lead-free. Small concentrations of aluminum in the bath are beneficial to reduce coating flaws when forging the coated parts. A hot dip bath with concentrations of aluminum exceeding fifty percent is reported to give "lifetime protection" to the underlying steel even under extreme conditions. Without the ability to confirm the elemental composition of the galvanization, it is difficult to warrant the materials' ability to perform as expected.

The LECO GDS500A is an atomic emission spectrometer that electronically records the spectra of each analysis. Elemental wavelengths can be defined within the analytical range of the spectrometer. The glow discharge source uniformly removes (sputters) material from the sample surface. Analysis takes place away from the sample surface reducing the effect of metallurgical history inherent in all samples. The excitation of primarily ground state atom lines means less complex spectra and reduced interferences than other AES sources. This is extremely important when base metal concentrations are equal or less than the bulk of the major elements. Calibration curves are mostly linear and cover a wide dynamic range. GD-AES is an excellent way to perform bulk chemical analysis on zinc bath samples.

Typical Analysis Results

TABLE 1: RESULTS OF ANALYSIS FOR NIST 1739
MATERIAL: ZINC - ALUMINUM ALLOY

ELEMENT	*RUN#1*	*RUN#2*	*RUN#3*	*AVERAGE*	*CERT.*	*% REL*	*STDEV*	*RSD*
Al %	0.21	0.20	0.20	0.20	0.20	0.26	0.002	1.20
Pb %	0.029	0.029	0.027	0.028	0.030	5.85	0.001	3.61
Zn %	99.76	99.77	99.77	99.77	—	—	—	—

TABLE 2: RESULTS OF ANALYSIS FOR MBH 43XZ2-E
MATERIAL: ZINC/ALUMINUM/COPPER ALLOY

ELEMENT	*RUN#1*	*RUN#2*	*RUN#3*	*AVERAGE*	*CERT.*	*% REL*	*STDEV*	*RSD*
Al %	4.15	4.16	4.18	4.17	4.20	0.81	0.017	0.40
Cd %	0.005	0.005	0.005	0.005	0.006	15.6	0.0003	6.03
Cu %	0.99	0.96	0.96	0.97	0.98	1.19	0.017	1.78
Fe %	0.038	0.037	0.038	0.038	0.038	1.14	0.001	1.77
Pb %	0.007	0.006	0.007	0.007	0.006	11.7	0.0004	5.97
Mg %	0.066	0.064	0.066	0.066	0.070	6.43	0.001	1.98
Sn %	0.007	0.005	0.005	0.006	0.006	1.67	0.001	19.3
Ni %	0.023	0.022	0.021	0.022	0.020	8.67	0.001	3.13
Zn %	94.72	94.74	94.71	94.72	—	—	—	—

Spectroscopy Performance Note

TABLE 3: RESULTS OF ANALYSIS FOR MBH 43XZ23
MATERIAL: ZINC/ALUMINUM/COPPER ALLOY

ELEMENT	*RUN#1*	*RUN#2*	*RUN#3*	*AVERAGE*	*CERT.*	*% REL*	*STDEV*	*RSD*
Al %	27.44	27.31	27.28	27.34	27.20	0.53	0.085	0.31
Cd %	0.012	0.012	0.011	0.012	0.011	6.97	0.0003	2.73
Cu %	2.97	3.04	3.00	3.00	3.07	2.23	0.032	1.07
Fe %	0.17	0.17	0.17	0.17	0.17	2.22	0.001	0.58
Pb %	<0.003	<0.003	<0.003	<0.003	0.002	—	—	—
Mg %	<0.005	<0.005	<0.005	<0.005	0.004	—	—	—
Mn %	<0.005	<0.005	<0.005	<0.005	0.002	—	—	—
Sn %	<0.005	<0.005	<0.005	<0.005	0.004	—	—	—
Zn %	69.40	69.47	69.54	69.47	—	—	—	—

TABLE 4: RESULTS OF ANALYSIS FOR HIGH ALUMINUM/ZINC ALLOY SAMPLE
MATERIAL: ALUMINUM - ZINC ALLOY

ELEMENT	*RUN#1*	*RUN#2*	*RUN#3*	*AVERAGE*	*STDEV*	*RSD*
Al %	66.72	66.92	67.04	66.89	0.16	0.24
Cd %	0.006	0.006	0.005	0.006	0.0003	5.52
Cu %	0.011	0.011	0.011	0.011	0.0002	2.07
Fe %	0.29	0.27	0.28	0.28	0.012	4.14
Pb %	0.014	0.015	0.012	0.014	0.001	7.88
Mg %	<0.005	<0.005	<0.005	<0.005	—	—
Mn %	0.006	0.006	0.006	0.006	0.0003	4.64
Si %	0.95	0.91	0.88	0.92	0.036	3.92
Zn %	32.00	31.86	31.77	31.87	—	—

Sample Preparation

The samples were surface abraded using a 320-grit silicon carbide disk (PN 810-253-PRM) with water on a LECO GPX300BMI (PN 824-200-900).

Surface contaminants that potentially exist are removed and discarded during a sputtering period called "preburn" prior to integration.

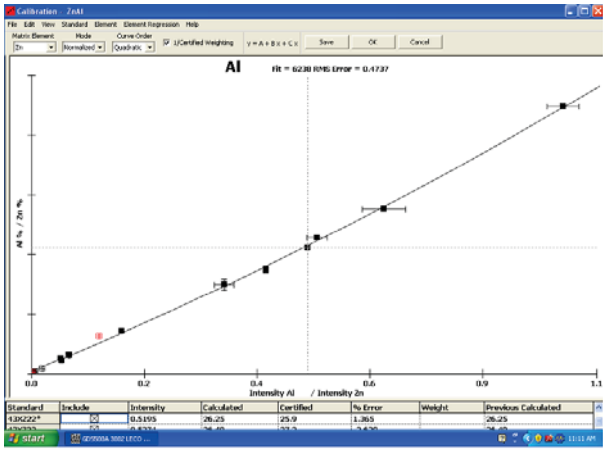
Accessories

LECO GPX300BMI, 320-grit silicon carbide disk.

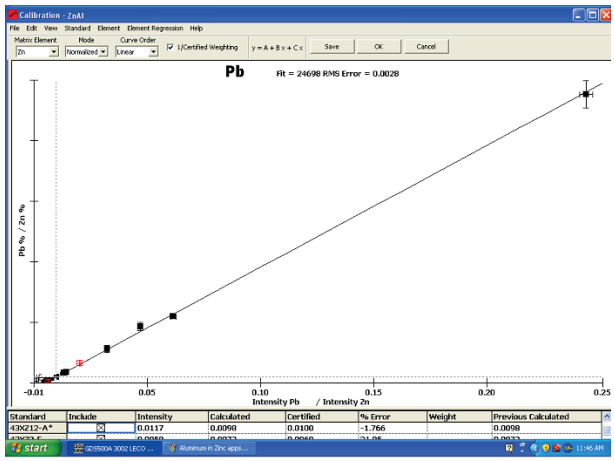
The GDS850A can perform bulk analysis on the bath as well as CDP analysis of the coated part. Direct analysis of the surface coating on the substrate can be performed using QDP software which records data from time zero through to the substrate. Using the GDS850A, coating thickness and weight can be determined quantitatively within minutes. Please reference performance note 209-076-027.

Calibration Curves

The high aluminum calibration curve shows a nearly linear fit from 5% at the lower section to over 50% at the top portion. A Certified Reference Material is shown plotted in the middle of the curve at 25% aluminum. In-house pieces have been added to prove accuracy at higher concentrations of aluminum in zinc bath. A more sensitive analytical line is used for lower concentrations of aluminum.



The lead curve is linear and accurate well below the 0.01%. Cross hairs indicate where the Certified Reference Material MBH 43XZ12-A falls on the calibration curve.



Calibration Standards

A factory-installed zinc alloy calibration is offered based upon specific customer requirements. Working curves are comprised of Certified Reference Materials (CRMs) and Reference Materials (RMs) and may include standards from the following manufacturers: NIST, Brammer, MBH, and ARMI. Customer supplied calibration pieces are useful to complement the calibration. Standards of high aluminum in zinc are commercially available however LECO cannot guarantee that they will be homogenous or fit for use.

Drift Control of Calibration

Homogenous non-certified set-up standards (SUSs) are used to drift correct calibration curves. When necessitated by customer ranges or lack of suitable SUS material, RMs and CRMs may be substituted.

Analysis Times

The LECO GDS500A has the ability to perform multiple analyses without dropping the sample. This is possible due to the sputtering away of material constantly revealing new, untouched sample for each analysis. Three replicate analyses can be completed in a minute and a half when using the "analyze all consecutive burns in one spot" option in the software.

	A single burn	*Three burns without dropping*
Start-up and Preburn	60 s	60 s
Analyze	10 s	10 s
Analyze		10 s
Analyze		10 s
Total	70 s	90 s

Spectroscopy Performance Note

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