Spectroscopy Performance Note

Bulk Analysis of Fasteners Using 2 mm Lamp (Rivet, Screw and Bolt)

Introduction

Fasteners such as bolts, screws, and rivets are modest fittings that are integral components of a majority of products we use and depend on everyday. Fasteners are expected to perform flawlessly for the entire lifetime of these products, which can range from one to over one hundred years.

Fasteners must therefore meet various physical requirements as well as chemical compositional specifications. The chemical composition of the fastener can affect mechanical properties, and gauge the level of future performance. It is prudent for the users of fasteners to conduct routine compositional testing to ensure that the fasteners conform to the intended chemical specification.

LECO Glow Discharge Spectrometers offer a way of determining the composition of samples as small as 2.5 mm in diameter. The data presented in this performance note are from fastener samples analyzed using the 2 mm lamp kit on a GDS500A. Such samples must bridge the anode/cathode gap and therefore need to be mounted in conductive media. For more details, please review the *Preparation of Specimens for Bulk Analysis Using LECO GDS* (209-076-031) application note at www.leco.com.

The LECO GDS500A offers you state-of-the-art technology designed specifically for routine elemental determination in most ferrous and nonferrous materials. LECO's exclusive CCD-based design ensures measurement stability, flexibility, and analytical performance in a production environment. GDS outperforms other excitation sources, because it uniformly removes (sputters) material from the sample surface and the analysis takes place away from the sample surface, reducing the effect of metallurgical history inherent in all samples. Additionally, since GDS records the excitation of primarily ground state atom lines the spectra are less complex and interference is reduced. Countless materials can be analyzed since GDS calibrations are inherently linear and cover a wide dynamic range. Furthermore, with options like the 2 mm lamp kit, these benefits are applicable to samples like fasteners, which pose significant difficulties for alternative techniques.

GDS500A

Typical Analysis Results

RESULTS OF ANALYSIS FOR RIVET (4 mm diameter shaft)
MATERIAL: ALUMINUM

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD
Cr %	0.073	0.074	0.073	0.073	0.001	0.83
Cu %	2.94	3.00	2.99	2.98	0.031	1.04
Fe %	0.40	0.37	0.43	0.40	0.030	7.56
Mg %	0.45	0.44	0.47	0.45	0.014	2.98
Mn %	0.14	0.14	0.14	0.14	0.003	2.30
Si %	0.35	0.36	0.36	0.36	0.005	1.41
Zn %	0.15	0.14	0.15	0.15	0.008	5.14
Al %	95.48	95.48	95.39	-	-	-

RESULTS OF ANALYSIS FOR SCREW (4 mm diameter threaded shaft)
MATERIAL: CARBON STEEL

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD
C %	0.39	0.38	0.38	0.39	0.006	1.58
Cr %	0.62	0.58	0.56	0.59	0.033	5.58
Cu %	0.027	0.022	0.023	0.024	0.003	12.9
Mn %	0.76	0.76	0.76	0.76	0.002	0.23
Mo %	0.23	0.22	0.23	0.22	0.003	1.29
Ni %	0.39	0.39	0.37	0.38	0.012	3.12
P %	0.027	0.028	0.028	0.028	0.001	2.31
Si %	0.30	0.30	0.29	0.29	0.006	2.20
S %	0.027	0.027	0.026	0.027	0.001	3.05
Al %	0.025	0.025	0.025	0.025	0.0002	0.83
Fe %	97.21	97.27	97.32	97.27	95.45	-

RESULTS OF ANALYSIS FOR BOLT (6 mm diameter threaded shaft)
MATERIAL: STAINLESS

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD
Al %	0.15	0.14	0.15	0.15	0.001	0.58
B %	0.002	0.002	0.002	0.002	0.0002	8.18
C %	0.041	0.041	0.043	0.042	0.001	2.91
Cr %	14.73	14.56	14.54	14.61	0.10	0.71
Cu %	0.14	0.13	0.14	0.13	0.003	2.59
Mn %	1.66	1.67	1.67	1.67	0.004	0.24
Mo %	1.25	1.25	1.22	1.24	0.013	1.07
Ni %	24.32	24.39	24.66	24.46	0.18	0.73
P %	0.023	0.020	0.020	0.021	0.002	9.09
S %	<0.003	<0.003	<0.003	<0.003	-	-
Si %	0.21	0.21	0.21	0.21	0.002	0.71
Ti %	2.21	2.21	2.21	2.21	0.002	0.09
V %	0.30	0.30	0.30	0.30	0.002	0.63
Fe %	54.97	55.07	54.84	54.96	-	-

Sample Preparation

Section the sample to give the largest surface area for analysis (for example, cut off the head of a screw or bolt and analyze where the shaft was attached.) Alternatively, press the shaft of the sample in a hydraulic press to flatten the specimen, thereby increasing the sample area. Mount in a conductive media as needed.

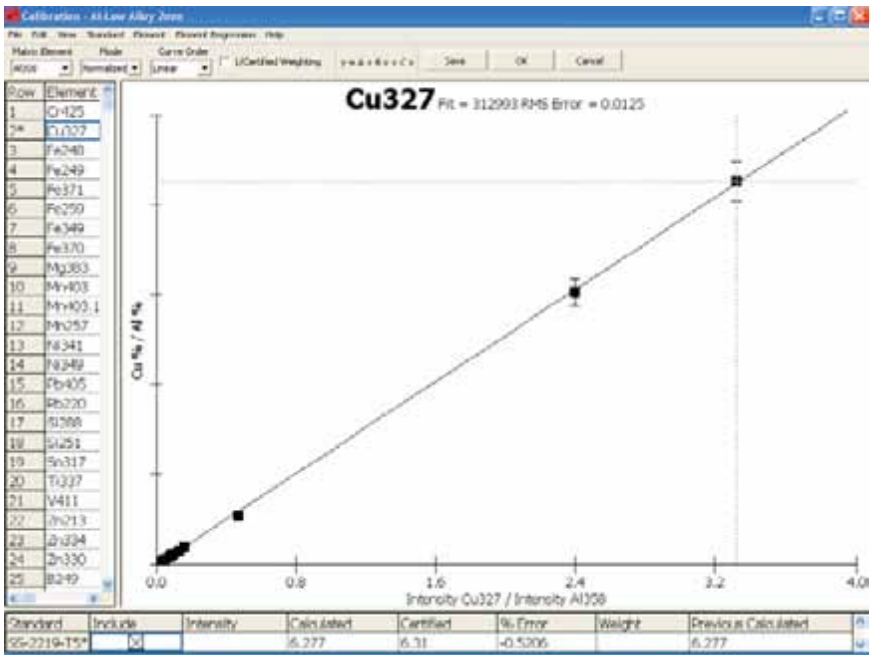
Ferrous-based samples mounted in copper diallyl phthalate are prepared using a 120-grit zirconium oxide belt or wet disk. A 320-grit wet finish is used for nonferrous matrices.

Accessories

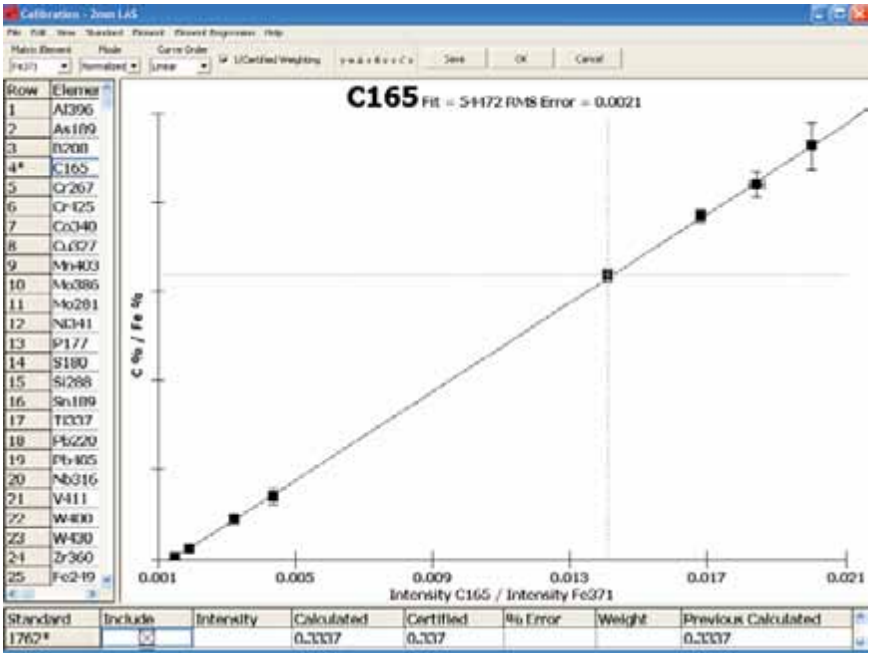
Sample surface preparation: Belt grinder (LECO BG) or polisher (LECO VP); Metallographic mounting press; Copper filled diallyl phthalate.

Calibration Curves

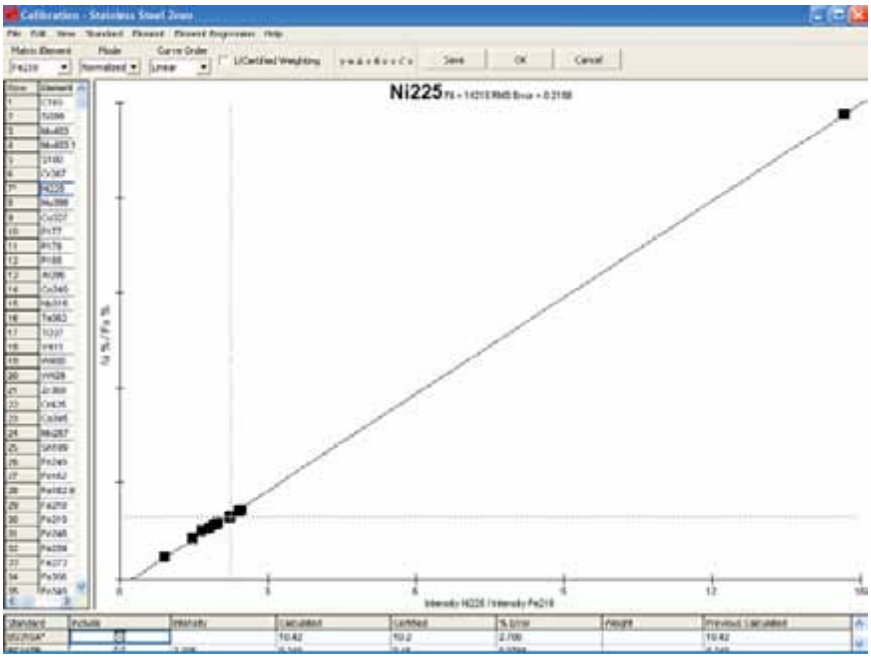
GDS calibration curves are linear over a large concentration range. The copper curve at right shows a very good fit through all the various aluminum alloys.



The carbon curve (right) demonstrates a very good fit with low alloy steels and carbon steels.



This nickel curve in stainless steel (right) demonstrates linearity through all of the stainless steel standards used for calibration.



Calibration Standards

A factory-installed calibration is offered based upon specific customer requirements. Working curves are comprised of Certified Reference Materials (CRM's) and Reference Materials (RM's) and may include standards from the following manufacturers: Alcoa, Alcan, NIST, MBH, CKD, BAS, Brammer, ARMI, VAW, and CTIF. Customer supplied calibration pieces are useful to complement the calibration.

Drift Control of Calibration

Homogenous non-certified set-up standards (SUS's) are used to drift correct calibration curves. When necessitated by customer ranges or lack of suitable SUS material, RM's and CRM's can be substituted.

Analysis Times

The LECO GDS500A has the ability to perform multiple analyses without dropping the sample. Three analyses can be completed in ninety seconds (compared to seventy seconds for one analysis) when using the "analyze all in one spot" option in the software. This is possible since the actual analysis occurs away from the surface and the sputtering process continuously reveals fresh unsputtered sample material for each analysis.

	A single burn	Three burns without dropping
Start-up and Preburn	60 sec.	60 sec.
Analyze	10 sec.	10 sec.
Analyze	—	10 sec.
Analyze	—	10 sec.
Total	70 sec.	90 sec.

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