Spectroscopy Performance Note

Routine Analysis of "Difficult" Alloys Using LECO Glow Discharge

Preface

The LECO GDS500A is an atomic emission spectrometer that electronically records the spectra of each analysis. All wavelengths can be defined within the analytical range of the spectrometer.

Glow Discharge routinely handles sample matrices that are difficult to analyze using other spectroscopic sources such as SPARK and X-Ray. Here are just a few of the difficult sample matrices that can be analyzed with a LECO GDS system.

- As-cast iron and ductile cast irons—able to quantify graphitic carbon
- Re-sulfurized and leaded alloys such as 12L14 steel and 303 grade stainless steel
- Nonferrous matrices such as leaded bronze and hypereutectic Al/Si alloy >15% Si
- Samples with high elemental levels of S and P as well as B and Mg are calibrated with no additional methodology
- Special alloys such as silicon carbide in aluminum

Another advantage is that GDS can analyze very small parts such as fasteners, washers, and screws. Small diameter wire including dental wires, A286 ribbon, and springs have been successfully tested. Powder samples that are malleable can be pressed into a pellet and analyzed directly on the GD lamp.

Why does Glow Discharge excel where other sources fall short?

The glow discharge source uniformly removes (sputters) material from the sample surface. The sputtered "crater" left behind is perfectly cylindrical with a flat bottom. This is evidence of the precise lamp control that allows sputtering to occur perpendicular to the anode. The sample never experiences a "melt pool" over which the spark randomly wanders and volatile elements are exposed to high temperatures. In GDS the relatively cool glow of the plasma samples material directly in its path. Contaminates at the surface are sputtered away during the pre-burn cycle revealing fresh unsputtered sample material. Surface preparation is therefore less of a problem with GDS than with spark and x-ray. In glow discharge the actual analysis takes place away from the sample surface that strongly reduces the effect of metallurgical history inherent in all samples. The excitation of primarily ground state atom lines means less complex spectra and sharply reduced interferences. Calibration curves are linear and cover a wide dynamic range. It is for these reasons that GDS is an excellent technique to perform chemical analysis on even the most difficult materials.

GDS500A

Typical Results

RESULTS OF ANALYSIS FOR BRAMMER STANDARD BS21A-G
MATERIAL: GREY CAST IRON

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
C %	3.88	3.88	3.84	3.87	3.83	0.96	0.023	0.60
Si %	1.55	1.55	1.58	1.56	1.56	0.085	0.016	1.02
Mn %	1.18	1.18	1.19	1.18	1.18	0.11	0.003	0.27
Mo %	0.19	0.19	0.19	0.19	0.19	1.43	0.001	0.35
S %	0.015	0.017	0.017	0.016	0.018	9.26	0.001	6.72
P %	0.075	0.077	0.075	0.076	0.070	8.33	0.001	1.85
Cu %	0.23	0.23	0.23	0.23	0.23	1.67	0.0004	0.16
Cr %	0.10	0.10	0.10	0.10	0.10	2.25	0.0003	0.33
Ni %	0.20	0.20	0.20	0.20	0.19	4.44	0.001	0.34
V %	0.016	0.015	0.016	0.016	0.016	2.50	0.001	3.57
Ti %	0.016	0.015	0.015	0.015	0.014	10.2	0.0003	1.63
Al %	0.021	0.021	0.021	0.021	0.020	6.17	0.0001	0.27
Fe %	92.53	92.52	92.53	92.53	-	-	-	-

RESULTS OF ANALYSIS FOR BRAMMER STANDARD BS284C
MATERIAL: CHILLED DUCTILE CAST IRON

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
C %	3.81	3.79	3.79	3.79	3.80	0.19	0.012	0.31
Cr %	0.070	0.070	0.072	0.071	0.073	3.38	0.002	2.29
Cu %	0.15	0.15	0.15	0.15	0.15	2.64	0.001	0.62
Mg %	0.014	0.017	0.015	0.015	0.015	2.00	0.001	8.19
Mn %	0.43	0.43	0.45	0.43	0.46	5.79	0.011	2.63
Mo %	0.010	0.010	0.007	0.009	0.008	8.33	0.0014	16.7
Ni %	0.046	0.046	0.046	0.046	0.045	2.67	0.0002	0.43
P %	0.031	0.032	0.029	0.030	0.031	2.15	0.001	4.89
S %	0.010	0.009	0.009	0.009	0.009	3.70	0.0004	3.76
Si %	1.78	1.79	1.76	1.78	1.83	2.75	0.014	0.80
Fe %	93.66	93.66	93.68	93.66	-	-	-	-

RESULTS OF ANALYSIS FOR IARM STANDARD 183A
MATERIAL: 12L14 STEEL ALLOY

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
C %	0.072	0.071	0.070	0.071	0.072	1.02	0.001	1.69
Cr %	0.011	0.011	0.011	0.011	0.010	6.67	0.0001	0.54
Cu %	0.006	0.005	0.005	0.005	0.006	12.2	0.001	12.2
Mn %	1.03	1.02	1.02	1.02	1.01	1.32	0.006	0.56
Mo %	<0.003	<0.003	<0.003	<0.003	0.002	-	-	-
Ni %	0.005	0.005	0.005	0.005	0.006	15.0	0.0003	5.19
P %	0.069	0.070	0.071	0.070	0.070	0.005	0.001	1.21
Pb %	0.23	0.23	0.22	0.22	0.24	6.94	0.005	2.07
S %	0.31	0.30	0.31	0.31	0.31	1.61	0.002	0.57
Fe %	98.27	98.29	98.29	98.29	-	-	-	-

RESULTS OF ANALYSIS FOR BRAMMER STANDARD BS80F
MATERIAL: STAINLESS STEEL GRADE 303

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
Al %	<0.002	<0.002	<0.002	<0.002	0.001	-	-	-
C %	0.061	0.059	0.059	0.060	0.062	3.92	0.001	2.25
Co %	0.16	0.16	0.16	0.16	0.16	1.06	0.0005	0.28
Cr %	17.06	17.26	17.10	17.14	17.10	0.23	0.11	0.62
Cu %	0.41	0.42	0.42	0.42	0.41	1.46	0.002	0.36
Mn %	1.77	1.77	1.77	1.77	1.76	0.53	0.004	0.20
Mo %	0.49	0.49	0.49	0.49	0.48	2.44	0.002	0.45
Nb %	0.016	0.014	0.014	0.015	0.016	9.17	0.001	8.03
Ni %	8.63	8.55	8.56	8.58	8.58	0.012	0.043	0.50
P %	0.032	0.032	0.032	0.032	0.036	10.6	0.0001	0.18
S %	0.34	0.34	0.34	0.34	0.35	2.13	0.002	0.49
Si %	0.62	0.62	0.65	0.63	0.63	0.35	0.019	3.02
Ti %	0.004	0.004	0.003	0.003	0.004	15.8	0.001	17.1
V %	0.084	0.083	0.086	0.084	0.087	3.26	0.002	1.79
W %	0.046	0.049	0.049	0.048	0.047	2.34	0.002	3.30
Fe %	70.27	70.14	70.26	70.22	-	-	-	-

RESULTS OF ANALYSIS FOR ARMI STANDARD 10A
MATERIAL: STAINLESS STEEL GRADE 416

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
Al %	<0.002	<0.002	<0.002	<0.002	0.002	-	-	-
C %	0.12	0.12	0.12	0.12	0.12	0.19	0.001	0.60
Co %	0.029	0.031	0.029	0.029	0.030	2.00	0.001	3.45
Cr %	12.24	12.23	12.24	12.24	12.18	0.47	0.006	0.05
Cu %	0.13	0.13	0.13	0.13	0.13	1.31	0.001	0.87
Mn %	0.81	0.80	0.79	0.80	0.80	0.30	0.009	1.13
Mo %	0.12	0.12	0.12	0.12	0.12	1.00	0.0007	0.58
Ni %	0.40	0.40	0.39	0.40	0.40	0.40	0.005	1.16
P %	0.024	0.024	0.024	0.024	0.024	1.11	0.0002	0.88
S %	0.37	0.38	0.37	0.37	0.37	0.89	0.003	0.75
Si %	0.53	0.53	0.52	0.53	0.53	0.62	0.002	0.41
V %	0.039	0.039	0.040	0.039	0.040	2.00	0.001	1.79
W %	0.012	0.013	0.013	0.013	0.013	3.08	0.001	7.27
Fe %	85.17	85.19	85.20	85.19	-	-	-	-

RESULTS OF ANALYSIS FOR CTIF STANDARD B32
MATERIAL: LEADED BRONZE

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
Pb %	16.04	16.10	16.09	16.08	16.15	0.45	0.032	0.20
Al %	0.125	0.125	0.126	0.125	0.125	0.16	0.001	0.50
Sb %	0.13	0.13	0.12	0.13	0.13	3.28	0.001	0.61
Fe %	0.14	0.14	0.14	0.14	0.13	5.74	0.001	0.71
Ni %	1.50	1.51	1.51	1.50	1.50	0.31	0.003	0.17
P %	0.045	0.047	0.047	0.046	0.047	1.13	0.001	2.18
S %	0.026	0.026	0.026	0.026	0.027	3.46	0.0002	0.80
Si %	0.094	0.093	0.094	0.094	0.095	1.19	0.001	0.71
Sn %	6.05	6.02	6.01	6.03	6.12	1.54	0.017	0.29
Zn %	1.40	1.40	1.40	1.40	1.41	0.66	0.002	0.15
Cu %	90.50	90.52	90.53	90.51	-	-	-	-

RESULTS OF ANALYSIS FOR MBH STANDARD 55XG28J5-P
MATERIAL: Al/Si/Cu HYPEREUTECTIC ALUMINUM

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	CERT	% REL	STDEV	RSD
Cu %	0.88	0.88	0.94	0.90	0.88	2.25	0.031	3.47
Mg %	0.55	0.55	0.58	0.56	0.52	7.51	0.016	2.92
Si %	23.85	23.58	22.21	23.21	23.20	0.06	0.88	3.79
Fe %	0.42	0.43	0.45	0.43	0.45	4.44	0.017	4.02
Mn %	0.76	0.76	0.77	0.76	0.76	0.09	0.006	0.84
Ni %	0.61	0.62	0.62	0.62	0.60	2.97	0.006	0.95
Ti %	0.19	0.19	0.19	0.19	0.19	0.88	0.001	0.29
Pb %	0.17	0.17	0.20	0.18	0.18	0.81	0.014	7.61
Al %	72.58	72.82	74.05	73.15	-	-	-	-

Sample Preparation

The LECO GDS500A and GDS850A spectrometers both come standard with an electronic help manual that includes a sample preparation guide. Typically, ferrous materials are prepared using a 120-grit zirconium oxide belt or wet disk. Nonferrous materials are prepared using a 320-grit silicon carbide paper on a wet disk. As-cast materials are finish prepared to a 600-grit finish.

Accessories

Sample surface preparation: Belt Grinder (LECO BG) or polisher (LECO VP).

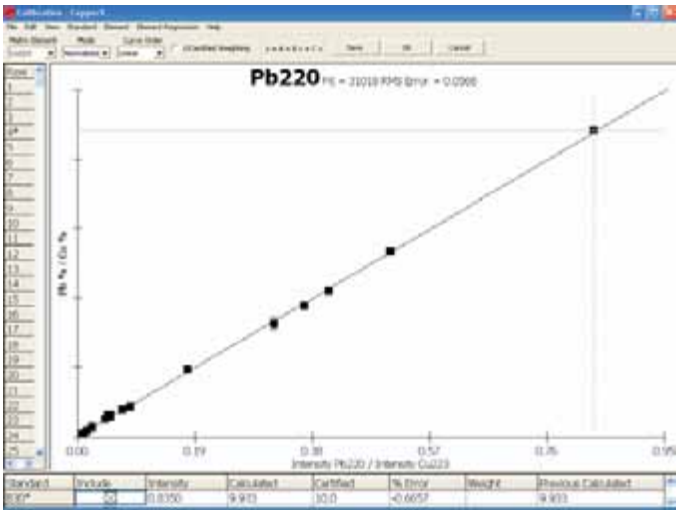
Calibration Curves

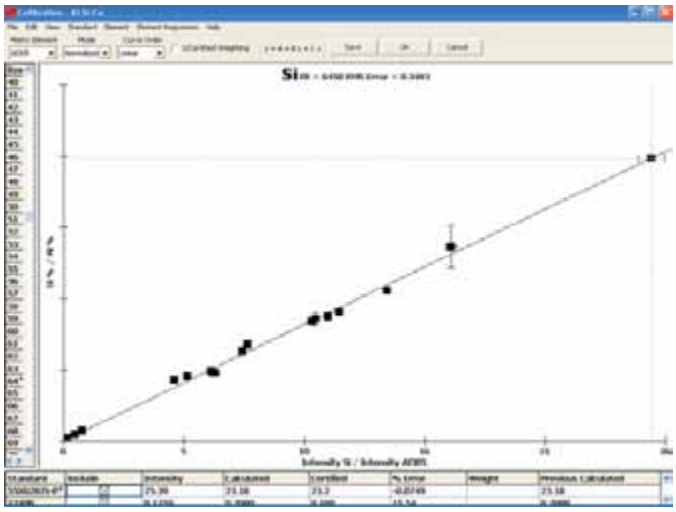
GDS calibration curves are inherently linear over a large concentration range. The lead curve (top right) shows a linear fit extending to 10% Pb. Linear calibration curves are accurate well beyond the last point on the calibration.

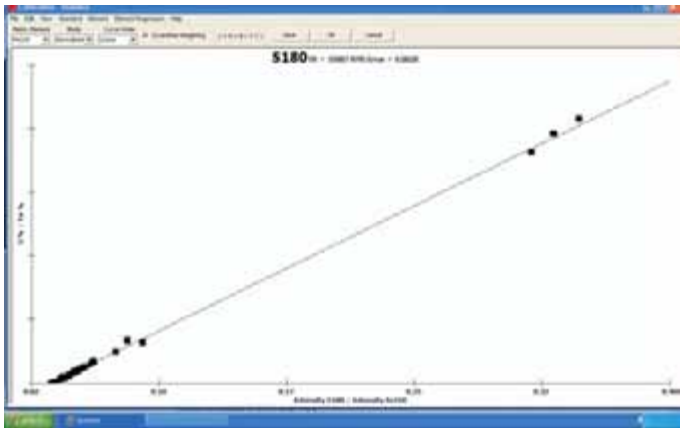
The silicon curve (middle right) demonstrates the linearity of a GDS calibration in wrought, cast, and hypereutectic grades of aluminum extending from 1 to 23% Si.

The sulfur curve (bottom right) demonstrates the glow discharge lamp's advantage over other sources when analyzing volatile species. The GDS source does not melt the sample surface. Another great advantage over other spectrographic sources is that the lamp is immune to contamination, meaning that there is no carry-over from one sample type to another.

continued on page 5

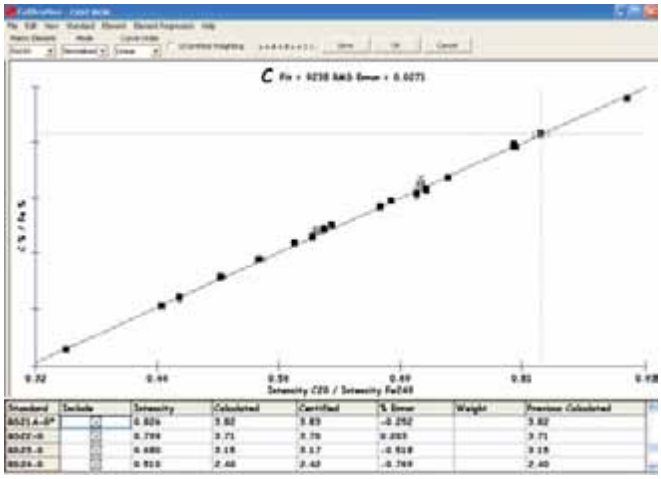






Calibration Curves *continued from page 4*

The carbon calibration curve (right) shows a very linear fit through all the cast iron standards which include chilled and as-cast (grey) types.



Calibration Standards

A factory-installed steel 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: NIST, Brammer, BAS, CTIF, MBH, Alcoa, Alcan, and ARMI. 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.[1]	60 sec.[1]
Analyze	10 sec.	10 sec.
Analyze	—	10 sec.
Analyze	—	10 sec.
Total	70 sec.	90 sec.

[1]*As-cast material requires a 180-second pre-burn time minimum.*

Additional Information

Additional information regarding sample preparation can be found in the GDS Sample Preparation Guide (Form No. 209-076-031), which can be found at **www.leco.com** (resources/applications library/spectroscopy).

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