

Spectroscopy Performance Note

Quality Control of Grade Specific Materials

Comparing the Small 2 mm and Standard 4 mm Glow Discharge Lamps (Grades 4340 & 8620)

Preface

One of the primary roles of a spectrometer is material grade confirmation. In this note we will compare data generated on a LECO GDS500A using two different size lamps, to illustrate how each is equally capable of grade specific analysis.

There are two lamps that can be used with the GDS500A; the regular 4 mm lamp and the optional small 2 mm lamp. The regular 4 mm lamp can accommodate a 2 mm inside diameter anode, whereas the small 2 mm lamp is specifically designed to use a thinner walled 2 mm anode. With previous systems, the user had to physically make a comparison of the analytical data to the material specification to determine whether the material was in or out of specification. This was a daunting task considering the variety of grades analyzed on a daily basis and sheer number of elements to compare.

To aid the user in the decision making process, the LECO GDS500A software allows input of user defined quality control limits. After each analysis, the results are automatically compared to the defined limits and flagged with a green star if acceptable, or with a yellow triangle if outside the inputted range. Additionally, the software allows the user to review multiple analyses in a table or graphical format that can be easily transmitted, printed, or stored.

The GDS500A is an atomic emission spectrometer with numerous advantages over competitive techniques. Since 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 inter-element interferences, both are extremely important when analyzing alloy materials. These features allow for extremely linear calibration curves that cover a wide dynamic range.

Typical Analysis Results

The grade specifications of interest were entered into the software as "System Limits" and known materials of 4340 and 8620 were analyzed. Figure 1 is a screen shot that shows the Limit Properties for 4340. Note the green star that appears in the row number indicating the elemental analysis has been compared to the limit and passed. A yellow triangle would appear if the limit was not met, with outlying analysis results in red. Tables 1 and 2 illustrate 4340 data for the small 2 mm and the regular 4 mm lamps. Tables 3 and 4 provide 8620 data for the small 2 mm and the regular 4 mm lamps. Please note that the analysis area of the 2 mm anode is not half but rather closer to one-quarter of the regular 4 mm anode.



GDS500A

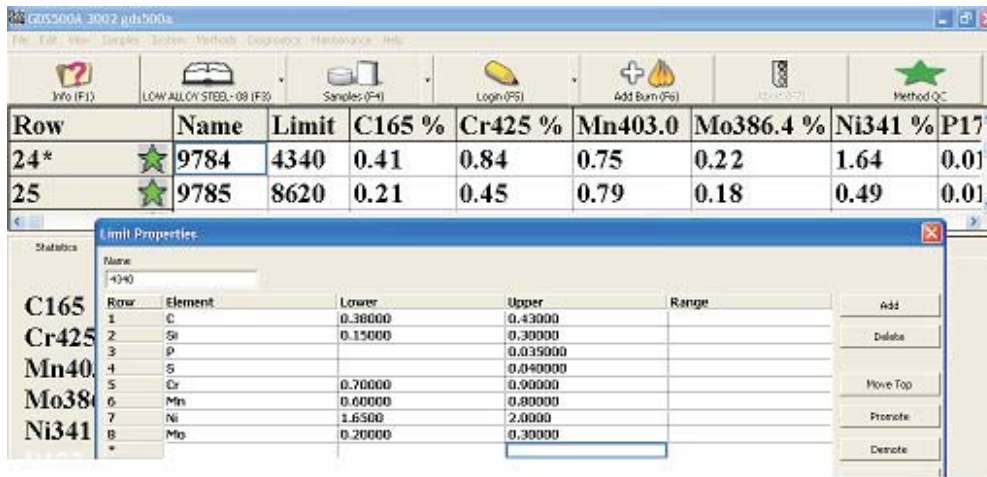


Figure 1: GDS500A Software screen shot showing Limit Properties and green star meaning the analysis has met the limits for this grade of steel.

**TABLE 1: SMALL 2 mm GDS LAMP
RESULTS OF ANALYSIS FOR SAMPLE 2674-9784 • MATERIAL GRADE: AMS 4340**

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD	SPECIFICATION	
							LOW (min)	HIGH (max)
C %	0.42	0.42	0.41	0.42	0.005	1.24	0.38	0.43
Cr %	0.86	0.86	0.86	0.86	0.002	0.18	0.70	0.90
Mn %	0.77	0.76	0.76	0.76	0.003	0.33	0.60	0.80
Mo %	0.22	0.22	0.21	0.22	0.004	2.03	0.20	0.30
Ni %	1.61	1.61	1.61	1.61	0.003	0.19	1.65	2.00
P %	0.011	0.010	0.011	0.011	0.0004	3.75	—	0.035
Si %	0.24	0.24	0.24	0.24	0.001	0.55	0.15	0.30
S %	0.023	0.023	0.024	0.023	0.001	3.31	—	0.040
Fe %	95.85	95.86	95.87	95.86	—	—	—	—

**TABLE 2: REGULAR 4 mm GDS LAMP
RESULTS OF ANALYSIS FOR SAMPLE 2674-9784 • MATERIAL GRADE: AMS 4340**

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD	SPECIFICATION	
							LOW(min)	HIGH(max)
C %	0.40	0.41	0.42	0.41	0.008	1.84	0.38	0.43
Cr %	0.84	0.84	0.84	0.84	0.00002	0.003	0.70	0.90
Mn %	0.75	0.74	0.75	0.75	0.003	0.45	0.60	0.80
Mo %	0.22	0.22	0.22	0.22	0.002	0.74	0.20	0.30
Ni %	1.65	1.66	1.63	1.64	0.014	0.83	1.65	2.00
P %	0.011	0.012	0.012	0.012	0.0009	7.41	—	0.035
Si %	0.22	0.21	0.21	0.21	0.001	0.60	0.15	0.30
S %	0.027	0.027	0.027	0.027	0.00004	0.13	—	0.040
Fe %	95.88	95.87	95.88	95.88	—	—	—	—

**TABLE 3: SMALL 2mm GDS LAMP
RESULTS OF ANALYSIS FOR SAMPLE 2674-9785 • MATERIAL GRADE: AMS 8620**

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD	SPECIFICATION	
							LOW(min)	HIGH(max)
C %	0.19	0.19	0.19	0.19	0.002	1.12	0.18	0.23
Cr %	0.44	0.44	0.44	0.44	0.0033	0.745	0.40	0.60
Mn %	0.79	0.79	0.78	0.79	0.007	0.84	0.70	0.90
Mo %	0.17	0.18	0.17	0.17	0.002	1.20	0.15	0.25
Ni %	0.47	0.48	0.48	0.48	0.002	0.41	0.40	0.70
P %	0.008	0.009	0.012	0.010	0.002	21.0	—	0.035
Si %	0.24	0.24	0.25	0.24	0.005	1.89	0.15	0.35
S %	0.025	0.023	0.020	0.023	0.003	12.21	—	0.040
Fe %	97.67	97.65	97.66	97.66	—	—	—	—

**TABLE 4: REGULAR 4 mm GDS LAMP
RESULTS OF ANALYSIS FOR SAMPLE 2674-9785 • MATERIAL GRADE: AMS 8620**

ELEMENT	RUN#1	RUN#2	RUN#3	AVERAGE	STDEV	RSD	SPECIFICATION	
							LOW(min)	HIGH(max)
C %	0.21	0.21	0.21	0.21	0.002	0.76	0.18	0.23
Cr %	0.45	0.45	0.45	0.45	0.00002	0.01	0.40	0.60
Mn %	0.80	0.80	0.79	0.79	0.002	0.26	0.70	0.90
Mo %	0.18	0.18	0.18	0.18	0.0005	0.27	0.15	0.25
Ni %	0.49	0.49	0.49	0.49	0.0005	0.09	0.40	0.70
P %	0.010	0.011	0.012	0.011	0.0009	8.23	—	0.035
Si %	0.22	0.22	0.22	0.22	0.0005	0.20	0.15	0.35
S %	0.023	0.023	0.024	0.023	0.0004	1.54	—	0.040
Fe %	97.63	97.63	97.63	97.63	—	—	—	—

Sample Preparation

Steel samples shall be prepared to a 120-grit or finer finish. A zirconium oxide belt was used on a LECO BG60 belt grinder. Contamination remaining after the grinding step is sputtered away during the preburn leaving a fresh, untouched layer from which the analysis data is taken.

Accessories

LECO BG60; 120-grit ZrOx belt (PN 810-499).

Working Curves

Working curves with the LECO GDS500A are linear over a large concentration range. The curves do not quit or “cut off” at the highest or lowest concentrations on the curve. Good accuracy can be expected at concentrations greater than 1.5 times beyond the highest calibration points so analytical results will be provided past these “end” points.

Calibration Standards

Working curves are comprised of Certified Reference Materials (CRM's) and Reference Materials (RM's) and may include standards from the following manufacturers: BAS, Brammer, MBH, NIST and ARMI. Customer supplied analyzed specimens are frequently useful to complement the calibration.

Control of Working Curves

Homogenous non-certified set up standards (SUS's) are typically used to drift correct working curves. We refer to this process as drifting or drift correction but it is also known as standardization.

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.

Additional Information

The main reason for choosing a 2 mm lamp vs. the 4 mm lamp is the size of sample predominately analyzed and the required o-ring seal. Sample sizes and ways to prepare the sample for analysis are discussed in great detail in the following performance note: [http://www.leco.com/resources/application_note_subs/pdf/spectroscopy/Performance Notes/209-076-031.pdf](http://www.leco.com/resources/application_note_subs/pdf/spectroscopy/Performance%20Notes/209-076-031.pdf). It should help determine which anode and lamp will best fit your specific needs.



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