Application Note

LECO EMPOWERING RESULTS

Instrument: GDS900

Using Bulk Analysis to Characterize the Carbon Gradient in Carburized Steel

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Key Words: Surface Carbon Analysis, Sub-Surface Analysis, Case Analysis by Grinding, Compositional Analysis, Glow Discharge AES, Glow Discharge OES

Preface

This application note demonstrates how the LECO® GDS900 can be utilized to characterize carburized steel using the standard low alloy steel method for bulk analysis. First, an unprepared surface is analyzed to a depth of approximately 100 µm. Next, successive layers are removed by "precision" grinding 100 µm at a time and performing analysis on each freshly revealed surface. Lastly, after reaching the base metal, analysis of the bulk material is performed.

Introduction

Carburization is a common surface treatment that enriches the carbon concentration in steel from the surface to a specific depth based on engineered specifications. The carbon-rich zone, or case, can range from the near surface to multiple millimeters in thickness. Temperature and hold time are parameters used to obtain the required carbon level and case depth. Knowing the initial steel composition is critical, especially the carbon concentration, since this affects the absorption and diffusion of carbon in the case. Physical tests, such as hardness and cross-sectional microscopy, confirm that carburization has been performed and provides the thickness or depth of the case.

The carbon composition at a given depth or thickness cannot be obtained with physical tests. This requires a carbon-specific analytical technique. Sampling for traditional elemental analysis techniques, such as combustion, can be difficult since milling or sectioning subjects the specimen to contamination, which causes erroneous results. The carbon gradient is simply too thin to be extracted in a precise way without potentially altering the carbon content. It is therefore more accurate and precise to determine carbon in place. Glow discharge AES can be utilized to analyze carburized steel directly, ensuring that the entire process is controlled from verification of the base alloy through quantitative analysis of the carbon rich gradient in the final product.

Glow discharge, in general, can sputter a sample to a maximum depth between 80–120 µm. When the crater becomes too deep, the plasma becomes unstable and self-extinguishes. A typical carburized case depth exceeds the crater depth that can be achieved during a maximized analysis cycle. Therefore, a more appropriate technique for revealing the carbon gradient is grinding away successive layers, followed by analysis of each freshly revealed surface.

Instrument Model

The LECO GDS900 is an atomic emission spectrometer that determines the elemental content of solid conductive materials by measuring the intensity of characteristic light emitted from the sample when excited. The glow discharge source uniformly removes (sputters) material from the sample surface, outperforming other excitation sources. Excitation of the atoms occurs in the glow discharge plasma discretely apart from the sample surface thereby reducing the metallurgical and chemical history inherent in all samples. Neutral atomic emission lines predominate the glow discharge spectra. While singly ionized transitions are observed in the glow discharge, the spectra are notably less complex than those produced by most other atomic emission techniques, resulting in few spectral interferences. In addition, the response of the typical glow discharge analytical line is linear and thus fewer wavelengths are required to determine the full analytical range.

The GDS900 offers 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.

Accessories

Sample preparation: Belt grinder (LECO BG Series) or polisher (LECO PX Series).

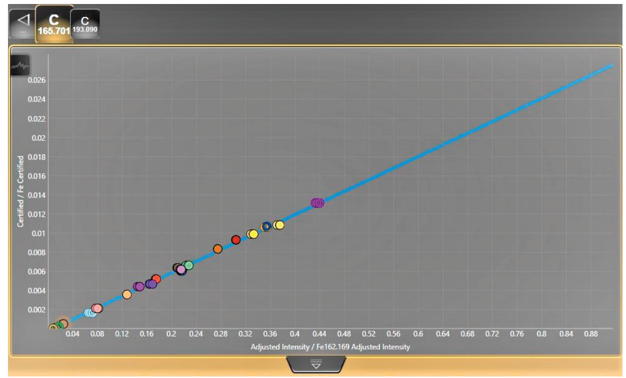
Sample Preparation

The sample was first analyzed with an unprepared (original) surface (refer to Figure 1). A belt grinder with 120-grit zirconium oxide belt (PN 810-499) was then used to prepare the sample for analysis (refer to Figure 2).

Note: If a belt grinder is not available then a polisher equipped with a 12 in disk (PN 812-046) with water or 8 in disk of 220-grit zirconium oxide (PN 812-024) with water can be used.

Calibration Curve

Carbon calibration curve with linear fit.



Calibration Standards

A factory-installed calibration based on each customer's distinctive requirements. Working curves are comprised of Certified Reference Materials (CRM's) and Reference Materials (RM's) from the following manufacturers: NIST, Brammer, CKD, MBH, and IARM. 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 are suitable replacements.

Analysis Times

The LECO GDS900 has the unique ability to perform multiple analyses without dropping the sample. This is possible due to the sputtering away of material to reveal new untouched sample. Three analyses are completed in a minute and a half when using the "Analyze all consecutive burns in the same spot" option in the software. *NOTE: This function was used extensively during the surface analysis where fifty replicates were analyzed (see Figure1).*

	Single Burn	**Three Burns w/o Dropping**
Start-up and Pre-burn	60 s	60 s
Analyze	10 s	10 s
Analyze	—	10 s
Analyze	—	10 s
Total	**70 s**	**90 s**

Surface Carbon Investigation

A graph of the surface carbon results from a carburized steel sample analyzed on the GDS900 is shown in Figure 1. The Low Alloy Steel (LAS) bulk method was used to collect data. The method was set with the minimum preburn setting (10 s) and fifty consecutive analyses in one spot without dropping the sample. Each data point represents a 10-second integration (total 500 s). This plot represents the carbon concentration at the very surface of the case.

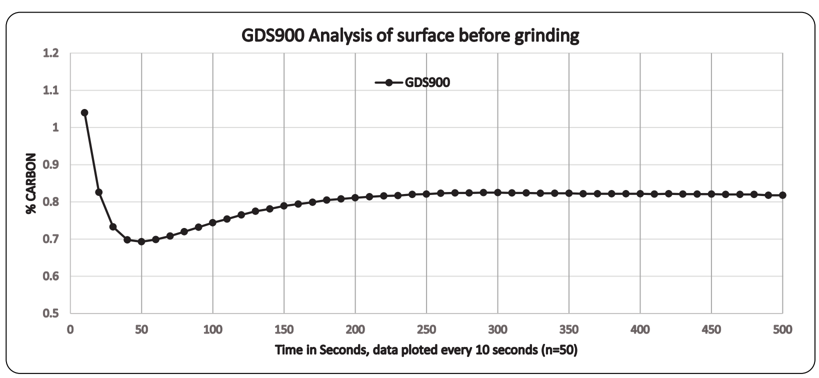


Figure 1. GDS900 percent carbon vs. time of the unprepared surface.

Revealing the Carbon Gradient in a Deep Case:

A graph of carbon concentrations through the case layer is shown in Figure 2. The data was collected using a technique referred to as "analyze-grind- analyze", where the sample is repeatedly ground then analyzed until the base metal is reached. Following grinding, three independent analyses were performed; the average is displayed on the plot. A digital caliper was used to measure the depth of material that was removed at each step. Typically, a specific layer of the case just below the surface is required to be tested; grinding to that exact depth can be accomplished quickly followed by analysis of the freshly revealed surface.

As can be seen in Figure 2, the carbon content decreases from a concentration of approximately 0.8% (which represents the initial case concentration shown in Figure 1) to the same concentration present in the bulk alloy of approximately 0.2% over a depth of 1100 μm. Eventually all the case was removed. When the carbon concentration stabilized, it matched the bulk concentration and could be included with the rest of the alloying constituents which make up the base metal. Table 1 presents the compositional analysis of the steel alloy including carbon.

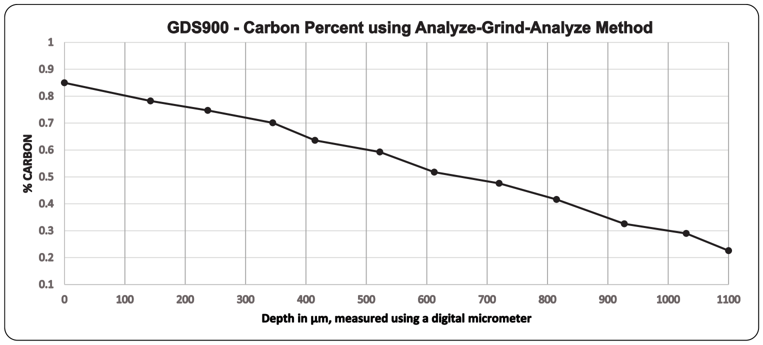


Figure 2. GDS900 percent carbon vs. µm of material removed from original surface.

Table 1. GDS900 bulk compositional data of base metal (carburized layer has been removed)

	C	S	Cr	Co	Cu	Mn	Mo	Ni	P	Si	Al	B	Fe
1	0.201	0.019	0.720	0.015	0.190	0.93	0.148	0.163	0.001	0.121	0.0249	0.0008	97.46
2	0.199	0.020	0.717	0.016	0.190	0.93	0.149	0.165	0.002	0.121	0.0249	0.0007	97.40
3	0.200	0.017	0.721	0.018	0.186	0.95	0.144	0.163	0.003	0.123	0.0246	0.0009	97.40
Average	0.200	0.018	0.719	0.016	0.189	0.94	0.147	0.164	0.002	0.122	0.0248	0.0008	97.42
Std.Dev.	0.001	0.002	0.002	0.001	0.002	0.013	0.003	0.001	0.001	0.001	0.0002	0.0001	0.04

Conclusion

Glow discharge spectrometry is an optimal tool for measuring surface carbon composition in materials like carburized steel. The GDS900 can accurately determine carbon at every step of the carburization process:

- Compositional validation of the base metal prior to heat treatment
- Surface carbon (near surface) after carburization if required
- Carbon and other elemental determination at any depth or layer by precision grinding
- Post carburization analysis for verification of the alloy

The intended purpose of this application note is to focus on the GDS900. However, the following LECO instruments were used to verify and correlate these results independently: GDS850A, CS744, and AMH55.

*A complete characterization of the carburized layer can be found in the webinar presentation, "Analysis of Carburized Steels by Glow Discharge Spectroscopy: Carbon in the Hardened Case and More". **Click here**.*

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