Spectroscopy Performance Note

Quantitative Depth Profile (QDP) Analysis of Carburized Steel

- **Carbon Concentration Gradient**
- **Case Depth**
- **Alloy Variation**

Introduction

Case hardening, or carburization, of steel is performed to provide improved tribological characteristics at the surface of a steel part (particularly abrasion, impact, friction, wear, and lubrication). Several methods of carburization exist, but primarily plasma or gaseous sources of carbon are used today. Gas carburizing, for example, is performed in a furnace at elevated temperatures in a high carbon environment in order to allow the diffusion of carbon into the surface layers of the metal lattice. This carbon concentration gradient is influenced by carburizing temperature and time, type of heat treatment cycle, carbon potential of the furnace atmosphere, and the original composition of the steel.

Of particular interest to carburizers is the depth and chemical compositional of the hardened case. A unique solution to this problem is Glow Discharge Atomic Emission Spectrometry (GD-AES) that can be used to characterize the hardened case of carburized steel.

The LECO GDS850A GD-AES allows for the quantitative depth profiling of the carburized surface by determining the chemical composition of the sample layer-by-layer. Since the GDS850A uniformly sputters the sample surface in a non-preferential manner, accurate sampling is attained at known depths. When these results are combined with results from a LECO LV700AT and/or LECO LCR500 Hardness Tester, an investigator can correlate Vickers and/or Rockwell-type hardness to specific chemical compositions and thoroughly characterize the case.

Investigation of a Carburized Part

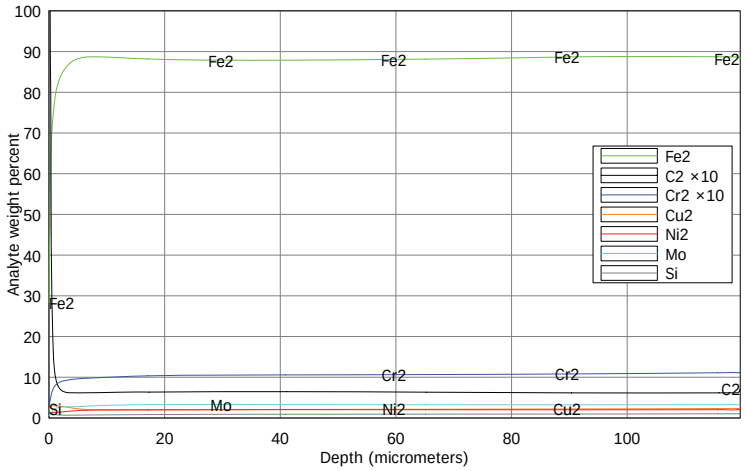
A carburized PW53 rod was analyzed on the GDS-850A for characterization of the hardened case. Analysis parameters are shown in Table 1.

Table 1: *Analytical Parameters for Carburized Steel*

Method Parameters	
Anode Diameter:	4 mm
Lamp Type:	DC
GDS operating Conditions:	40 mA, 1200 V
Discharge Stabilization:	Current (Control Mode)/Voltage (Pressure Control)
Minimum Data Acquisition Rate:	10 s^{-1}
Profile Duration:	1200 s, thickness dependant
Cooling:	Closed Cooling

GDS850A

Quantitative Depth Profile
Operator: DG
Notes: PW 53 Original Surface



Depth (µm)	C2 (%)
0.5	2.9
1.0	1.1
1.5	0.81
2.0	0.69
2.5	0.65
3.0	0.63
3.5	0.62
4.0	0.61
4.5	0.61
5.0	0.61

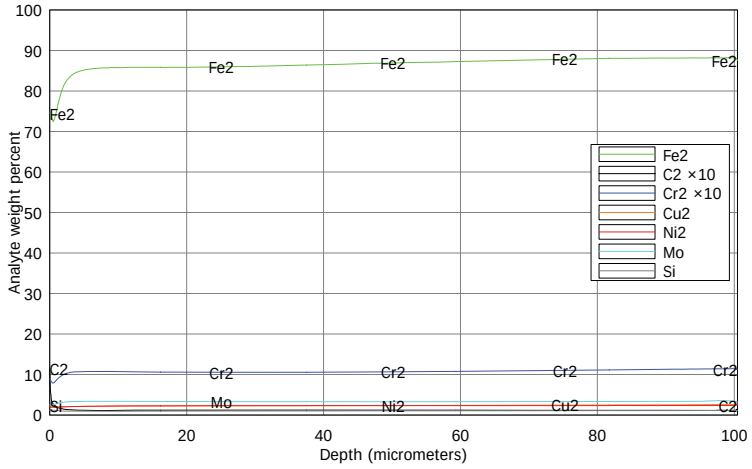
Depth (µm)	C2 (%)
5	0.61
10	0.63
15	0.63
20	0.64
25	0.64
30	0.64
35	0.64
40	0.65
45	0.64
50	0.64
55	0.64
60	0.64
65	0.63
70	0.63
75	0.63
80	0.62
85	0.62
90	0.62
95	0.61
100	0.61

Figure 1.

Quantitative Depth Profile (QDP) analysis was performed on the original sample surface and on eleven subsequent ground surfaces for a total analysis depth exceeding 2 mm. Each QDP was allowed to sputter until self extinction, typically 80 to 120 µm. Following analysis on each surface, material was removed past the depth of the sputter crater using a precision grinder and finished wet with 320-grit SiC paper on a LECO VP-50 Polisher/Grinder. Figure 1 shows a plot of analysis displayed in concentration in weight percent versus depth in micrometers (µm) of the original sample surface. The table(s) shows the carbon concentration gradient with respect to depth. The carbon value is shown every 0.5 µm for the first 5 µm and every 5 µm thereafter.

Figure 2 shows the innermost analysis at a depth of 2.28 mm. The carbon value (C-0.11%) is equal to the substrate concentration as confirmed by combustion analysis (C-0.11%) on a LECO CS600 Carbon/Sulfur Determinator.

Quantitative Depth Profile
Operator: DG
Notes: 2.28 mm



Depth (µm)	C2 (%)
5	0.12
10	0.12
15	0.12
20	0.12
25	0.12
30	0.12
35	0.12
40	0.12
45	0.12
50	0.12
55	0.12
60	0.12
65	0.12
70	0.12
75	0.11
80	0.11
85	0.11
90	0.11
95	0.11
100	0.11

Figure 2.

At each surface of analysis for the QDP analysis, hardness measurements were performed using both Vickers (HV)—LECO LV700AT Hardness Tester—and Rockwell "C" (HRC)—LECO LCR500 Hardness Tester. Table 2 shows hardness in HRC, HV, and HRC converted from HV (HRC(conv)), and carbon concentration versus depth in millimeters.

Figure 3 displays the variation in both hardness and carbon concentration versus depth, and Figure 4 shows the variation of hardness versus carbon concentration.

Table 2: Variation of hardness and carbon concentration versus depth

Depth(mm)	HRC	HV	HRC(conv)	C%
0.00	60.6	636	57.2	0.84
0.08	59.2	709	60.4	0.70
0.26	59.0	694	59.8	0.68
0.44	58.3	699	60.0	0.67
0.55	58.8	682	58.8	0.54
0.65	55.0	635	57.1	0.52
0.95	50.6	575	53.8	0.38
1.30	46.1	499	49.0	0.29
1.52	42.4	475	47.3	0.22
1.76	42.1	431	43.7	0.17
2.02	40.5	417	42.4	0.15
2.28	40.0	389	39.6	0.13

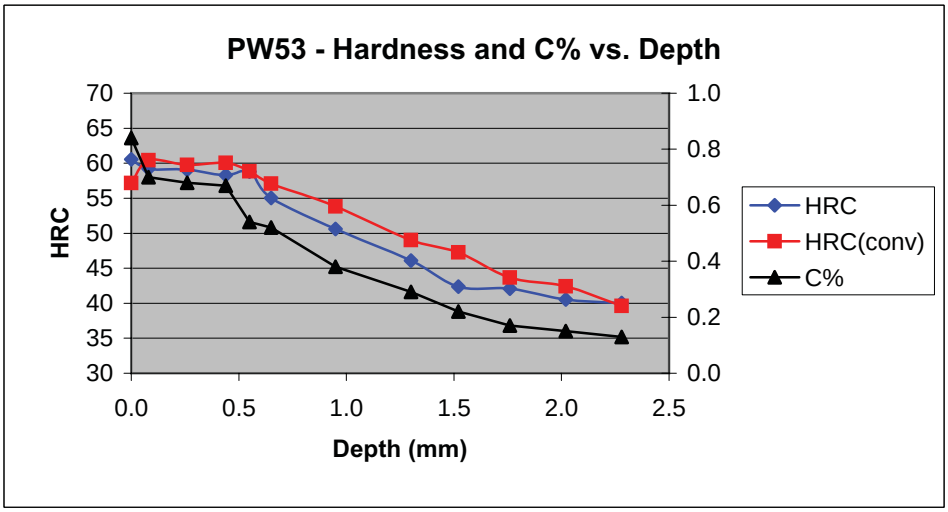


Figure 3: Variation of hardness and carbon concentration versus depth

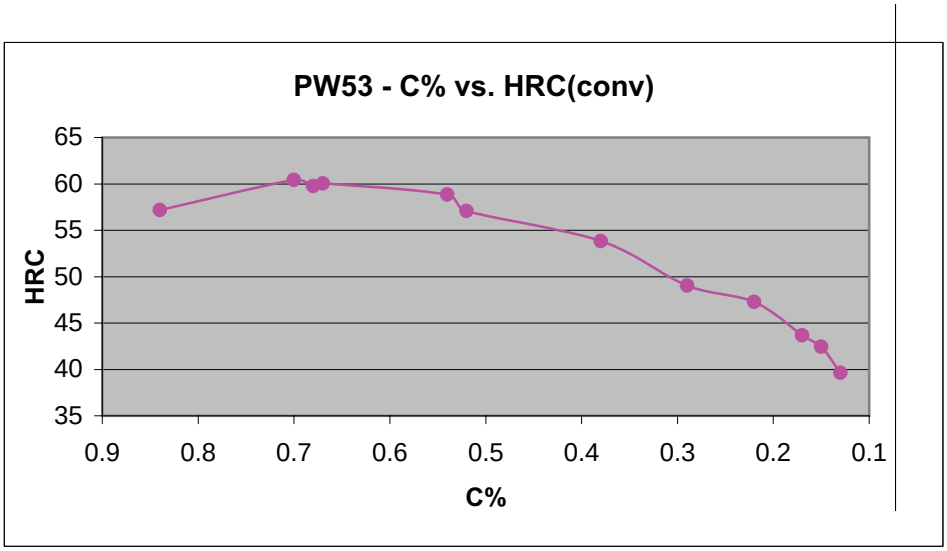


Figure 4: Variation of hardness versus carbon concentration

The PW53 rod was also sectioned on a LECO MSX250A Sectioning Machine, mounted in epoxy using a LECO PR-25 Mounting Press, and polished on a LECO GPX300 Grinder/Polisher using 0.05 μm colloidal silica as the final polishing step. The polished section was etched with 2% Nital and examined on a LECO GX51 Inverted Stage Metallograph with PAX-it™ Image Management System. Figure 5 is a metallographic image at 50X which clearly shows the case and the substrate microstructures.

Microhardness analysis was performed on the polished cross section using a Knoop (HK) indenter—LECO LM247AT—with a 500 g load on a LECO AMH43 Automatic Microindentation System. The results of analysis are shown in Figure 6 and include a table and three plots. The table to the left gives hardness in HK and HRC conversion versus depth in millimeters. The upper plot shows HK vs. depth, the middle plot shows HRC vs. depth and the lower plot shows HRC vs. the first 3 mm of depth.

Summary

Quantitative Depth Profile analysis provides case depth information in line with that found by cross sectional metallography. In addition, changes in alloy composition with depth can be determined simultaneously with carbon content.

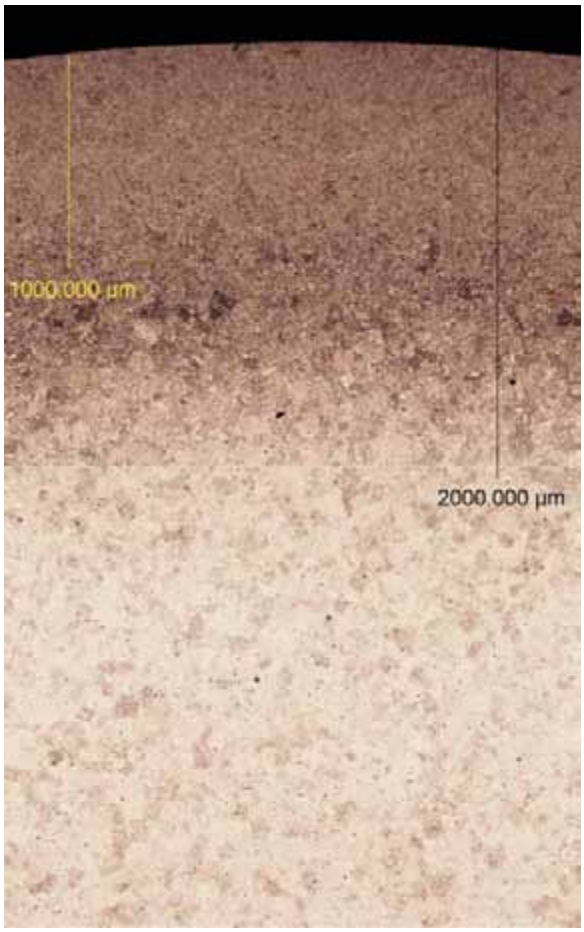
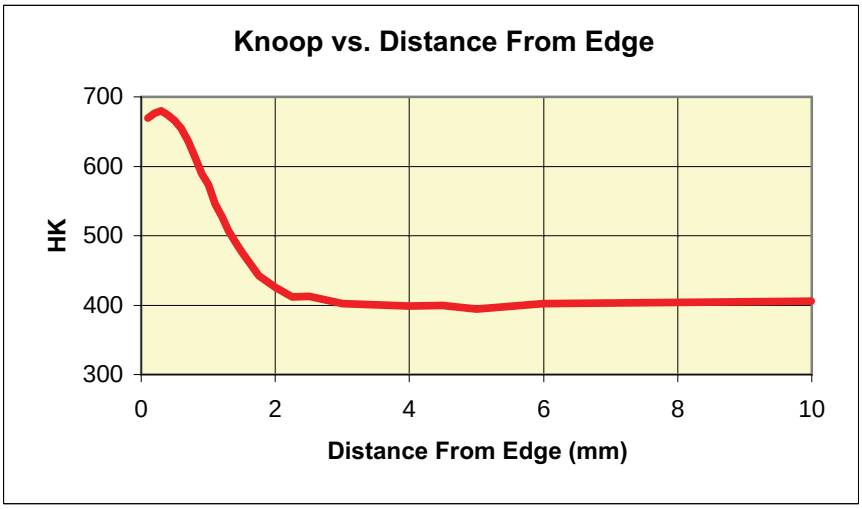
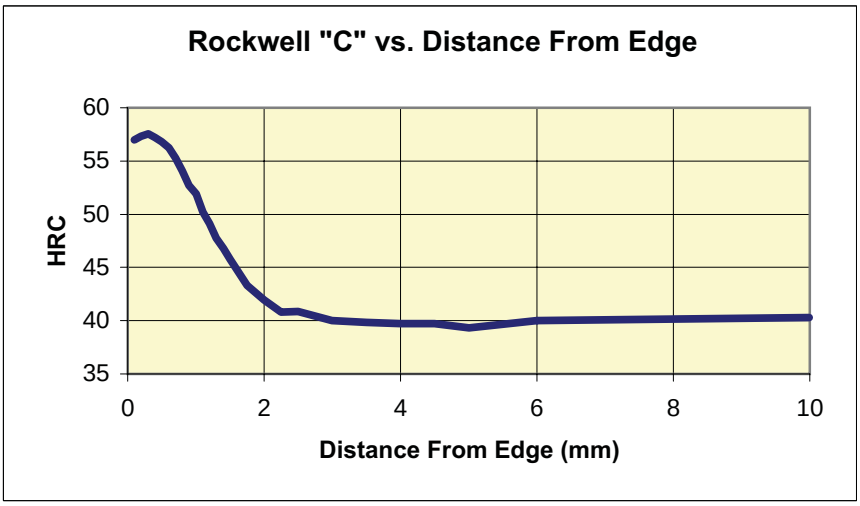


Figure 5: Cross sectional image of sample PW53 50X – 2% Nital

Averages of Four Traverses:

Distance	HK	HRC
0.10	669	57.0
0.20	677	57.4
0.30	680	57.6
0.40	674	57.2
0.50	666	56.8
0.60	654	56.2
0.70	636	55.3
0.80	613	54.1
0.90	589	52.7
1.00	574	51.9
1.10	546	50.2
1.20	528	49.1
1.30	507	47.8
1.40	492	46.8
1.50	477	45.8
1.75	443	43.3
2.00	426	42.0
2.25	412	40.8
2.50	412	40.9
3.00	403	40.0
3.50	401	39.8
4.00	399	39.7
4.50	399	39.8
5.00	395	39.3
6.00	402	40.0
10.0	406	40.3





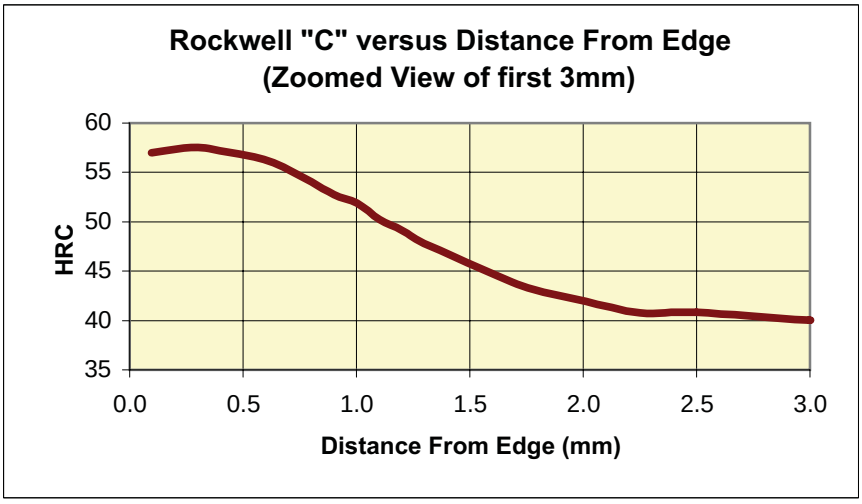


Figure 6: Knoop microhardness analysis on the AMH43 Automatic Microindentation System

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