Analysis of Trace Elements in Tungsten Carbide, Using a Barium Fluoride Carrier, on a Prodigy DC Arc Spectrometer

Introduction

Tungsten carbide is a fine gray powder with a melting point of 2870 °C and a hardness of 8.5-9.0, surpassed in hardness only by diamond materials. In its powdered form, tungsten carbide can be pressed into various shapes for use in tools, abrasives, jewelry, industrial drills, armor-piercing ammunition, spikes for snowmobile equipment and studs for bicycle tires. Tungsten carbide is predominantly used in combination with metallic cobalt to produce cemented carbide which is used to improve the strength, hardness and thermal stability of wear parts and mining tools. The result is increased lifetime and robustness for products such as industrial punches and hot rollers used for rolling steel.

This application note contains data to demonstrate the ability of the Teledyne Leeman Lab's **Prodigy DC Arc** to determine trace elements in high-purity tungsten carbide.

Experimental

Operating Parameters

All standards and samples were prepared for analysis by mixing each with a carrier consisting of a 50:4:1 blend of graphite, barium fluoride and germanium(IV) oxide. The standards and samples were mixed such that the ratio of standard/sample to carrier-containing graphite was 1:1. Each mixture was thoroughly blended with a SPEX mixer/mill for a minimum of 5 minutes before hand packing it into sample electrodes.

All samples were analyzed on the Teledyne Leeman Lab's **Prodigy DC Arc**. Standards and samples were burned in atmosphere and the remaining instrument and method conditions are listed in Table I.

Table I DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignition at 6A, hold for 2 s, jump to 10A, hold for 58 s
Stallwood Jet	None
Analytical Gap	4 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	3/16" diameter with an undercut cup (ASTM #S-15)
Sample	
Sample Size	Hand packed, ~40 mg
Internal Standard	Ge at 270.963 nm
Integration Time	0-60 s for all wavelengths

Sales/Support: 800-634-9942 • **Main**: 603-886-8400
110 Lowell Rd, Hudson, NH 03051
www.teledyneleemanlabs.com

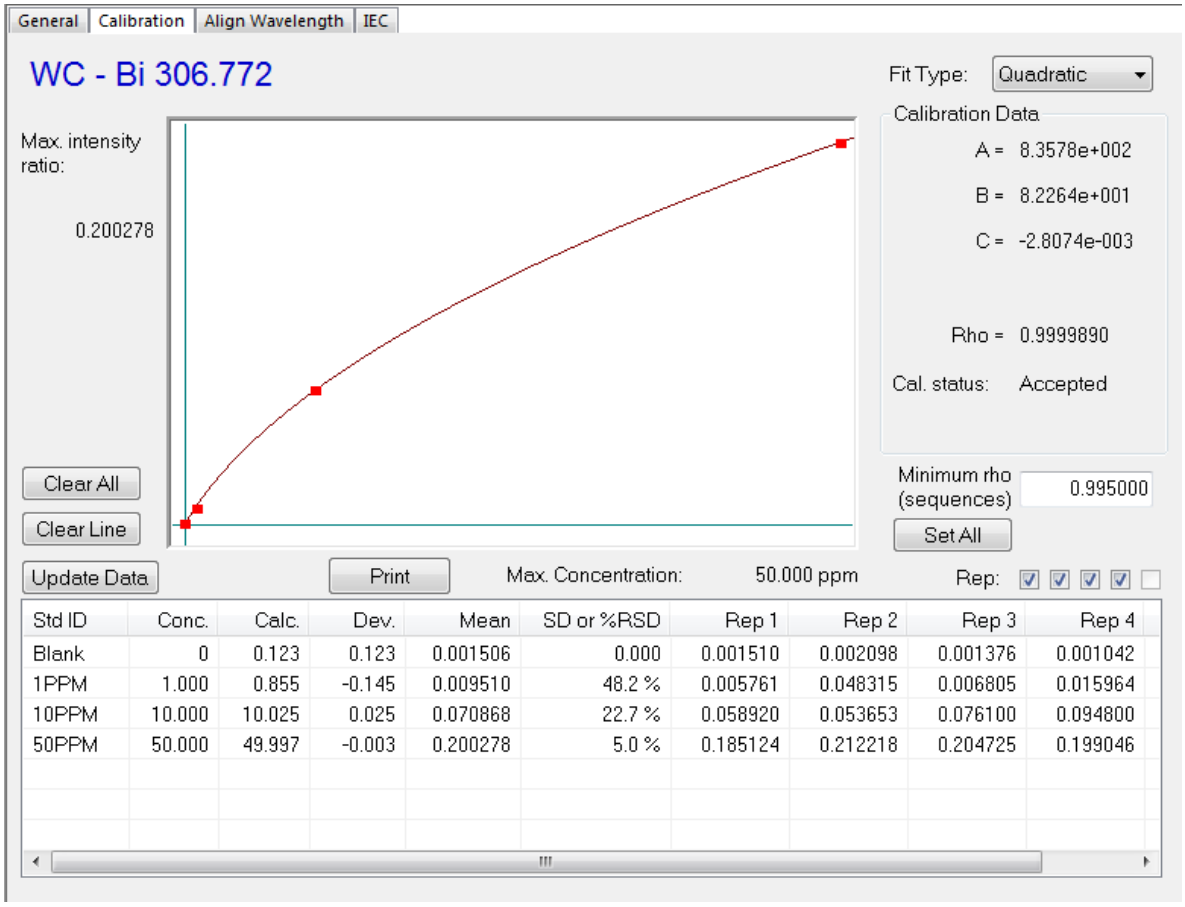
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with an undercut cup (part # S-15). The counter electrodes used were 1/8" in diameter and pointed (part # C-1). A 4 mm analytical gap was used and the position of the electrodes was adjusted during the sample burn to maintain a distance of 4 mm between the sample and the counter electrode.

Calibration

The instrument was calibrated with several high-purity tungsten metal standards that contained the analytes of interest at 0, 1, 10, 50 and 100 ppm. Tungsten metal was used for calibration instead of tungsten carbide because tungsten metal behaves similarly to tungsten carbide in a DC Arc, and tungsten metal is readily obtained at a higher purity. Calibration standards were prepared by serial dilution on a weight-to-weight basis from a multielement stock standard containing 45 elements at 1.21% (MV Laboratories, Inc., Frenchtown, NJ). All standards were weighed, mixed and prepared for analysis with graphite as described above.

An example calibration curve for elements measured in high-purity tungsten carbide is illustrated in Figure 1 for Bi at 306.772 nm. The calibration curve for Bi demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve for Bi at 306.772 nm in High-Purity Tungsten Carbide



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
Blank	0	0.123	0.123	0.001506	0.000	0.001510	0.002098	0.001376	0.001042
1PPM	1.000	0.855	-0.145	0.009510	48.2 %	0.005761	0.048315	0.006805	0.015964
10PPM	10.000	10.025	0.025	0.070868	22.7 %	0.058920	0.053653	0.076100	0.094800
50PPM	50.000	49.997	-0.003	0.200278	5.0 %	0.185124	0.212218	0.204725	0.199046

Results

Detection Limits

A study was performed to determine the instrument's detection limits for the elements of interest. Detection limits were calculated based on 3 times the standard deviation of 9 replicate measurements of the calibration blank. Results for the detection limit study are listed in Table II in units of parts per million (ppm). Since barium fluoride and germanium(IV) oxide were used in the sample preparation method, detection limits for Ba and Ge were not calculated.

It should be noted that the detection limit for Si reflects inhomogeneties in the standards more than imprecision in the instrument.

Table II Detection Limits in High-Purity Tungsten Carbide

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	328.068	0.046	0-60	Mn	260.569	0.020	0-60
Al	308.216	0.28	0-60	Mo	313.259	0.77	0-60
As	193.759	4.55	0-60	Na	330.232	1.84	0-60
B	249.678	0.062	0-60	Nb	309.418	0.89	0-60
Be	234.861	0.002	0-60	Ni	305.082	0.11	0-60
Bi	306.772	0.080	0-60	P	253.565	0.73	0-60
Ca	317.933	2.83	0-60	Pb	283.307	0.053	0-60
Ca	393.366	0.49	0-60	Sb	231.147	0.47	0-60
Cd	214.438	0.69	0-60	Se	203.985	2.68	0-60
Co	345.351	0.026	0-60	Si	251.921	3.53	0-60
Cr	284.325	0.062	0-60	Sn	317.502	0.19	0-60
Cu	327.396	0.025	0-60	Sr	407.771	0.22	0-60
Fe	259.940	0.29	0-60	Ti	334.941	0.093	0-60
Ga	294.364	0.021	0-60	Tl	535.046	0.41	0-60
K	766.491	0.29	0-60	V	318.540	0.32	0-60
Li	610.364	0.034	0-60	Zn	213.856	0.048	0-60
Mg	280.270	0.041	0-60	Zr	343.823	0.30	0-60

Conclusions

The analysis of tungsten carbide using the **Prodigy DC Arc** demonstrates that the current-controlled DC Arc power supply, combined with the simultaneous data collection of both peak and background data, provides reproducible sample burns that are reflected in the detection limits obtained for trace elements in a tungsten carbide matrix.