Analysis of Trace Elements in Silicon Metal Using the Prodigy DC Arc Spectrometer

Introduction

Silicon is a blue tinted metal with a high luster and a melting point of 1414 °C. It is the second most abundant element found in the Earth's crust, yet silicon is rarely found as a free element in nature. Due to its electrical resistance and relatively high thermal conductivity properties, silicon is used in a wide range of applications. The solar industry increasingly makes use of silicon in the production of silicon wafers, photovoltaic solar cells and silicon-based electronic semiconductors. As an alloyed metal, ferrosilicon accounts for a majority of all silicon produced worldwide, and is used in the steel industry. Silicon is also used to improve the hardness and wear-resistance of aluminum which makes it useful in the production of steels.

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 Si metal.

Experimental

Operating Parameters

All standards were prepared for analysis by grinding each sample to a fine powder in a tungsten carbide mixing vial with tungsten carbide mixing balls. The mixing vials were shaken in a SPEX mixer/mill for a minimum of 5 minutes prior to hand packing each sample into sample electrodes.

All analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc** with a 70:30 mixture of $Ar:O_2$ flowing through the Stallwood jet. The remaining instrument and method conditions used are listed in Table I.

Table I DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignition at 9A, hold for 2 s, jump to 12A, hold for 20 s, jump to 15A, hold for 93 s
Stallwood Jet	70:30 $Ar:O_2$
Analytical Gap	3 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	3/16" diameter with 4 mm x 3 mm undercut cup (ASTM #S-15)
Sample	
Sample Size	Hand packed
Internal Standard	None
Integration Time	0-115 s

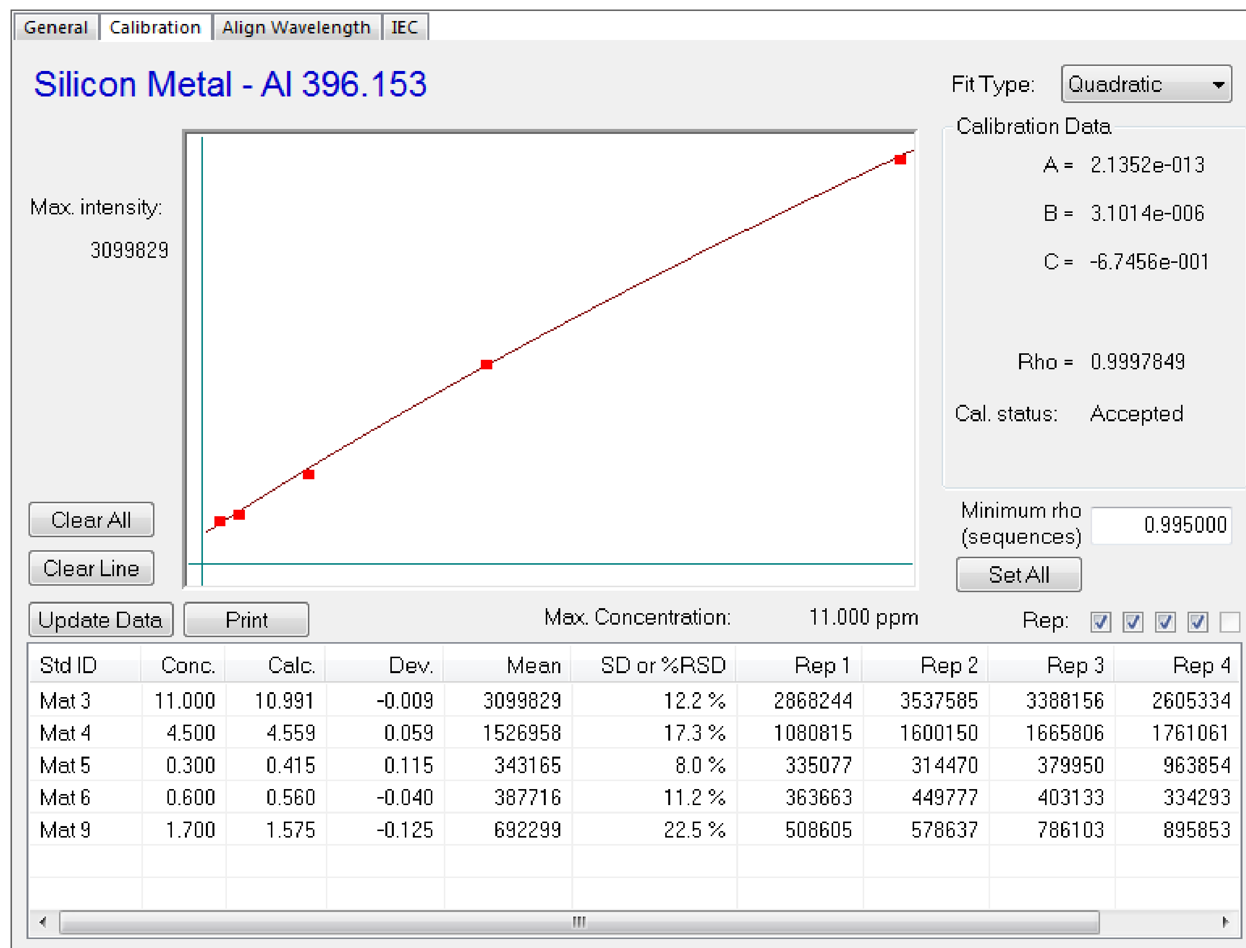
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with an undercut cup (part # S-15). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 3 mm analytical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

Calibration

The instrument was calibrated with several high-purity Si metal standards that contained the analytes of interest at concentrations that ranged from 0.05 to 2200 ppm. All standards were prepared for analysis as described above.

An example calibration curve for elements measured in Si metal is illustrated in Figure 1 for Al at 396.153 nm. The calibration curve for Al demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve of Al at 396.153 nm in High-Purity Silicon Metal



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
Mat 3	11.000	10.991	-0.009	3099829	12.2 %	2868244	3537585	3388156	2605334
Mat 4	4.500	4.559	0.059	1526958	17.3 %	1080815	1600150	1665806	1761061
Mat 5	0.300	0.415	0.115	343165	8.0 %	335077	314470	379950	963854
Mat 6	0.600	0.560	-0.040	387716	11.2 %	363663	449777	403133	334293
Mat 9	1.700	1.575	-0.125	692299	22.5 %	508605	578637	786103	895853

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 5 replicate measurements of the lowest calibration standard. Results for the detection limit study are listed in Table II in units of parts per million (ppm).

Table II Detection Limits in High-Purity Silicon Metal

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Al	396.153	0.34	0-115	Mn	279.827	1.4	0-115
B	249.773	1.1	0-115	Ni	300.249	0.74	0-115
Ca	393.366	0.67	0-115	P	213.618	5.3	0-115
Cr	427.480	1.9	0-115	Ti	336.121	0.56	0-115
Cu	327.396	0.49	0-115	V	318.540	0.56	0-115
Fe	302.064	3.0	0-115				

Conclusions

The analysis of Si metal 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 Si metal matrix.