SPECTRO **REPORT**

APPLICATION REPORT 63

SPECTRO Analytical Instruments GmbH

DETERMINATION OF MINOR AND TRACE ELEMENTS IN SILICON CARBIDE BY SLURRY SAMPLE PRESENTATION/ICP

Summary

A suite of six silicon carbide samples were submitted for analysis by the slurry/ICP technique. These were in the form of fine powders with a mean particle size of 3µm. Approximate analyses were given with three of the samples, the other three treated as unknowns. The samples were analyzed for the following eleven trace and minor constituents: Fe, Al, B, Cu, Ni, V, Na, Cr, Ca, Co and Ti.

Introduction

The analysis of materials such as silicon carbide is generally a challenge. The material is very refractory and requires a good deal of sample preparation for conventional ICP analysis. The ability to run these materials in slurry form would greatly reduce analysis time for this type of sample.

Operating Conditions

A standard SPECTRO purged sequential/simultaneous instrument with simultaneous internal standard capability was used. Hardware Requirements for Slurry Nebulisation:
- Continuous Sample Agitation
- Babington Nebuliser
- Direct Path Sample Presentation

Slurry/ICP Operating Parameters:

- Sample delivery rate:	2 ml min-1
- Nebuliser flow:	0.7 l min-1
- Auxiliary flow:	0.8 l min-1
- Coolant flow:	12 l min-1
- Observation zone:	15 mm above coil
- RF power:	1200 W

Sample Preparation

4 g of sample were weighed out and 40 ml of 0.1% v/v Triton X-100 in deionised water added. Samples and standards were stirred for 1 min prior to analysis.

Typical Sample Preparation Procedure For Slurries:
1. Grind to -325 (<45um) or -400 (<38um) mesh in mill - 10 min.
2. Weigh into disposable 50 ml beaker
3. Add 25 ml water
4. Add 1 drop of surfactant (Triton X-100)
5. Stir for 1 min.

Analytical Figures of Merit

Wavelength Scans

Scans of two preanalyzed samples are appended to this report. Signals observed from the silicon internal reference indicate that the 10% (w/v) silicon carbide slurry gave a signal level equivalent to about a 4-5% (w/v) solution of the same sample. The scans are shown with a dionised water blank, a spiked solution blank and the two samples. No major line overlaps were noted for any of the analyte lines. However background shifts can be noted for certain analyte lines such as V 292.402 nm and several potential weak line overlaps can be noted. Appropriate application of off peak background correction and line overlap correction would be necessary to preserve accuracy close to detection limit.

System Calibration

The system was calibrated using the three analyzed samples. The samples had previously been run by DC arc and these values were taken as actual concentrations. In the case of V the reported value was >360 ppm. Several weak silicon lines were evaluated and Si 256.864 nm was selected as internal standard. All readings were ratioed to this line to compensate for differences in sample particle size distribution. Several analytes did not have a wide enough range in concentration to allow calibration. Calibration curves are given for the following analyte lines (calibration curve for Ca is presented in fig. 1):

Fe	259.940
Al	308.215
Cu	324.754
Ni	231.604
V	292.402
Cr	267.716
Ca	317.933
Ti	334.941

Background Equivalent Concentrations and Detection Limit Values

In general all detection limits for the analyte elements studied are < 1 ppm (µg/g) in the solid.

Element	Line (nm)	BEC	DL
Fe	259.940	6	0.2
Cu	324.754	5	0.2
Ni	231.604	20	0.6
V	292.402	15	0.5
Cr	267.716	2	0.1
Ca	317.933	1.2	0.04
Ti	334.941	2.2	0.08

Precision

Precision values expressed in % relative standard deviation are given below for ten consecutive runs of STD-202. These are unratioed values. For concentrations above BEC it can be seen that precision is typically 1-2% RSD or better. Ratioing to the internal standard generally improves precision a further factor of two.

Element	Line (nm)	Precision (%RSD)
Si	256.864	1.3
Fe	259.940	1.3
Al	308.215	1.2
Cu	324.754	0.8
Ni	231.604	1.5
V	292.402	2.1
Cr	267.716	0.9
Ca	317.933	2.0
Ti	334.941	2.1

Results For Unknown Samples

Values for the three unknown samples are given below: Note that the V data should be treated with extreme caution as the high standard concentration was taken as > 360 ppm (µg/g).

Element	Sample No.		
	202 *(µg/g)*	*207* *(µg/g)*	*222* *(µg/g)*
Fe	4	460	70
Al	67	75	80
B	<1	<1	<1
Cu	<1	<1	<1
Ni	<1	<1	<1
V	11	(348)	(700)
Na	<1	<1	<1
Cr	2	18	18
Ca	4	31	32
Co	<1	<1	<1
Ti	14	56	76

Figure 1: Calibration curve for Ca 317.933 in Silicon Carbide by ICP/Slurry