CHAPTER 11

CONCLUSION

The research was undertaken to show whether:

a) an alternative, rapid dissolution method which avoids the use of reagents that have the potential to complicate the analysis could be found

b) the ICP-OES measurement technique is suitable for the measurement of cobalt, tantalum, titanium, vanadium and chromium in a tungsten carbide solution, and

c) accurate and precise results can be obtained with such a technique.

11.1 DISSOLUTION PROCEDURE

The dissolution procedure was proved effective for the dissolution of cobalt, tantalum, titanium, vanadium and chromium in tungsten carbide powders. The material was usually completely dissolved within 10 minutes with the hydrogen peroxide/ aqua regia method, compared to 45 minutes or more for the altemative nitric acid/ hydrofluoric acid method. The hydrogen peroxide/ aqua regia method utilizes less expensive high purity acids and the waste products present fewer problems than with traditional methods. Less expensive equipment is required, since standard laboratory glassware may used instead of PTFE vessels required for use with hydrofluoric acid.

11.2 ICP-OES MEASUREMENT

The measurement experiments proved that the ICP-OES technique is suitable for the measurement of the elements investigated, in a complex tungsten carbide matrix. The main advantage of ICP-OES over atomic absorption spectrometry is the fact that several analytical wavelengths may be evaluated during a single run, thus reducing the time required for analyses and well as the amount of work. The use of more than one analytical wavelength is a convenient

quality control aid, since interferences seldom occur to exactly the same extent at two different wavelengths. Any differences in analytical results at the different wavelengths may be used to indicate a problem.

Another advantage is the multi-element capabilities of ICP-OES. When different target elements are present in the samples in similar concentration ranges, the calibration solutions can be adapted to include them all.

A summary of the analytical wavelengths used for each element, as well as the coefficients of determination (r^2), and the limits of detection (LOD) and quantification (LOQ) achieved are given in Table 11.1.

Table 11.1: Summary of data obtained by ICP-OES measurements

Element	Concentration in sample (%)	Analytical wavelength (nm)	LOD_{soln} (mg/ ℓ)	LOD_{smpl} (g/ 100 g)	LOQ_{soln} mg/ ℓ	LOQ_{smpl} g/ 100 g	r^2
Co	1 - 15	228.616	2	0.4	7	1	0.9998
		238.346	1	0.2	4	0.8	0.9999
Ta	0 – 0.8	240.063	1	0.02	3	0.06	0.9993
		268.517	0.4	0.01	1	0.02	0.9999
Ti	0 - 4	337.280	0.3	0.01	1	0.02	0.9999
		368.520	0.2	0.004	0.7	0.01	0.9999
V	0 - 8	292.402	0.03	0.001	0.1	0.002	1.0000
		311.071	0.1	0.002	0.5	0.01	0.9999
Cr	0 – 0.2	357.869	0.2	0.004	0.6	0.01	1.0000

The ICP-OES technique was found to be rapid and convenient. It also offered more scope for

the investigation and detection of matrix related measurement problems than, for example, atomic absorption spectrometry.

11.3 ACCURACY AND PRECISION

The accuracy of the measurements was evaluated through the use of certified reference materials, secondary reference materials, matrix spiking and the method of standard additions. The ICP-OES results compared well to results obtained in other laboratories using XRF.

11.4 SHORTCOMINGS

The hydrogen peroxide could not be removed from the sample solutions prior to ICP-OES measurement without causing tungsten compounds to precipitate. This caused a slight, but acceptable increase in the RSD values obtained.

Because the cobalt content of the samples was 10 to 50 times higher than the concentrations of the other elements measured, cobalt had to be measured separately after further dilution. This caused an increase in the time taken for the analysis.

11.5 SPECIAL POINTS

The dissolution method described is a new approach to the problems encountered in the sample preparation for the analysis of tungsten carbide compounds.

No information was previously found on the analysis of vanadium in tungsten carbide by ICP-OES. The experimental results obtained show that excellent results can be obtained.

11.6 RECOMMENDATIONS AND FURTHER RESEARCH

The research was done in a commercial laboratory, where turnaround times and the limiting of costs are very important. The data presented was produced from the analysis of more than 100 samples of different compositions.

The exact minimum concentration of hydrogen peroxide required to keep the sample in solution should be studied in order to increase the precision of the results. A further systematic study of the selected wavelengths may show that a single analytical line for each element may produce consistently accurate results, no matter how the composition varies.

It would be helpful if certified reference materials of tungsten carbide, containing tantalum, titanium, vanadium and chromium in concentrations close to those of actual samples, could be obtained and used as quality control aids.