3. EXPERIMENTAL PROCEDURE

This chapter describes the methods used in this study. It starts with the preparation of the samples, characterization of the material, the methods and conditions used to apply thermal shock and the abrasive wear tests. Lastly, different microscopes used to analyze the features caused by thermal shock are described.

A WC-12wt%Co alloy was selected for this investigation, because it is a widely used mining grade [2,3]. BOART LONGYEAR supplied the samples. It is used in the mining industry as its excellent properties (good combination of hardness and toughness) make it suitable to withstand the harsh conditions. Refer to Figure 2.1, which shows SEM micrograph of the WC-12wt%Co microstructure.

3.1 Sample preparation

Samples of WC-12wt%Co were prepared by first cutting the samples to the required size, followed by mounting and polishing for microscopic viewing before and after thermal shock and abrasive wear tests.

The specimens were cut into 9×6×5 mm plates using a Struers Accutom cutting machine (with a diamond wheel). This specific size of the specimens was required to fit the specimen into the sample holder of the wear equipment. A Leco PR-25 mounting press and a thermosetting bakelite resin were used to mount the specimens. Specimens were then ground and polished using a BUEHLER METASERV universal polisher to obtain the same surface finish on all the specimens prior to performing the tests. Grinding was done under running water

using 80, 120, 220 and 1200 grit sizes. Polishing was done using 3µm and 1µm synthetic diamond sprays with lubricant fluid for extended time to prevent any generation of stresses.

Cross-sections were prepared by cutting the samples using a diamond saw. This was done before and after thermal shock to analyze the depth affected by the thermal shock. Samples were then polished to remove any features introduced by cutting.

3.2. Sample characterization

The hardness, WC grain size, Co mean free path and the density of the samples were measured as these properties influence the wear response of the hardmetals. The wear rate of the material with high density is faster as compared to material with a lower density.

3.2.1 Vickers hardness

Macro-hardness tests were carried out using a Leco V-100-A2 tester. The resolution of this instrument is ± 0.5HV. As reported in the literature review the hardness of the material affects the wear rate; the wear rate generally increases as the hardness decreases. The samples were polished to a 1µm finish prior to the test. Ten indentations were made on each sample with a 30kgf applied load.

3.2.2 WC grain size and Co mean free path

The WC grain size and the Co mean free path were determined using back-scattered electron micrographs taken with a Jeol JSM840 scanning electron microscope and an image analyzer (Wacom IntuosTM). Approximately 1000 WC grains were counted using a WACOM® click sensitive tablet connected to a computer. The statistical distribution of the WC grain size and the Co mean free path were determined using Microsoft Excel, and the average WC grain size and the mean free path values calculated.

3.2.3 Density

Density is expected to be low on hardmetals containing oxides and porosities, and this will result in high wear rate. The Archimedes principle was used to measure the density of the samples. The mass of the dry sample of WC-12wt%Co was measured. Then the samples were suspended in distilled water with a wire and weighed. The density was calculated using the formula below. Three samples were measured and the average was taken as the density of the WC-12wt%Co.

$$\rho = \rho_{water} \times \frac{Wt_{water}}{Wt_{air} - Wt_{water}}$$

Where: ρ_{water} = density of water

Wt_{air} = weight of specimen in air

Wt_{water} = weight of specimen in water

3.3 Thermal shock tests

A heat treatment was required in which only the surface of the specimen would be heated and then quenched. Various options were discussed, like using a high temperature lamp. But the safest and most viable method was selected based on the availability of the instruments and the practicable procedures. The specimen would be heated for an extremely short time period in a furnace in air and after each time period quenched in water.

A furnace was commissioned for this project (see Figure 3.1). The outer size of the furnace is 43.5×38.5×49.5 cm. The size of the heating chamber is 34×17×11.3 cm. The temperature inside the furnace is measured by means of a thermocouple. A maximum temperature of 1000ºC can be reached. This temperature is achieved in approximately 160 minutes (see Figure 3.2).

Figure 3.1: Furnace used for heat treatment.

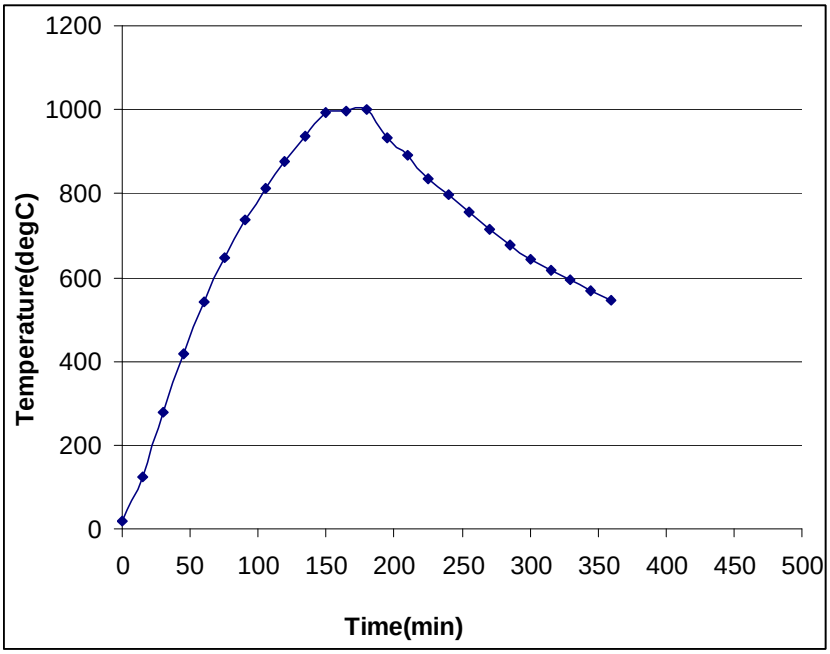

Figure 3.2: Calibration of the furnace used (heating and cooling rate).

Figure 3.2 is a temperature calibration graph: The furnace was set to a temperature of 1000ºC which was achieved after approximately 160 minutes, then allowed to cool down by switching it off and leaving the door closed. The rate of heating is faster than the rate of cooling. Calculations were required to determine the exposure time for the specimen in the furnace; not to heat the whole sample but only the surface. WC-Co alloys have good thermal conductivity implying that the material is a good conductor and will reach the temperature rapidly [1].

Prof A. Bryson, a specialist in heat transfer theory, derived the formula relating the temperature in the furnace, the time and the depth of the sample that was heated. The transfer of heat to the sample occurs via two mechanisms, namely conduction and convection. In conduction, heat is transferred directly from a warm material to a cooler material whereas convection is by movement of air in the furnace i.e. heat transfer by the circulation of air currents from one region to another. The formula derived was:

$$\theta(y,t) = 1 - erf\left(\frac{y}{2\sqrt{\alpha t}}\right) - \left[\exp\left(\frac{hy}{k} + \frac{h^2\alpha t}{k}\right)\right]\left[1 - erf\left(\frac{y}{2\sqrt{\alpha t}} + \frac{h\sqrt{\alpha t}}{k}\right)\right]$$

Where; $\theta(y,t)$ = the heat conducted during a time t through a depth y.

t = the time of exposure to heat

y = depth of heated sample

k = thermal conductivity,

α = thermal diffusivity

h = 10W/m^{2}K (Boltzmann's constant)

The complete derivation of this formula is given in Appendix A.

From Prof Bryson's formula, a very short heating time appeared to be required to limit the heating to the surface layer. It was calculated that <1sec is required to reach a depth of 100µm (y) at 800°C (θ). However as this time is too short for practical purposes (since the sample had to be put into the furnace and removed by hand), the best, shortest, feasible time was selected instead. 30 seconds was the heat treatment time that was selected for all the tests. Temperatures of 600°C, 800°C and 1000°C were selected based on the service temperatures of rock drilling inserts, which varies depending on the type of the rock being drilled [2]. The furnace was first heated up to required temperature. The samples were quenched in water at room temperature long enough to cool to room temperature, which was the temperature of the water. The duration of this quenching process was about one minute. The experiments done on this project do not directly simulate what is in industries, because of the complexity involved in industries.

Two sets of thermal shock tests were carried out. The first set consisted of keeping the temperature constant while varying the number of thermal shock

cycles; the second consisted of keeping the number of thermal shock cycles constant while varying the thermal shock temperature. See tables 3.1 and 3.2.

Table 3.1: Test conditions at constant thermal shock temperature of 800ºC.

Temperature in the furnace	800°C
Heat treatment time per cycle	30sec
Temperature of water	Room temperature, 23ºC
Quenching time	1min
Number of thermal shock cycles	60, 80 and 100 cycles

Table 3.2: Test conditions at constant thermal shock cycles (60 cycles).

Number of thermal shocks	60 cycles
Heat treatment time per cycle	30sec
Temperature of water	Room temperature, 23ºC
Quenching time	1min
Range of temperatures	600°C, 800°C and 1000°C

3.4 Abrasive wear tests

Abrasive wear tests were performed on 80-grit SiC abrasive papers using a 2-body sliding wear apparatus (see Figures 3.3 and 3.4). This 2-body sliding wear was an in-house instrument which was then modified by T.A Mokgalagadi (MSc. Student). The SiC abrasive paper has a diameter of 200mm. The instrument is designed so that the sample holder that is attached to the loading arm moves along a radius of the rotating abrasive paper.

The specimens were weighed prior to the wear test. During the wear tests, the sample moves along a radius towards the center of the SiC paper while the paper rotates, so that the path described by the sample relative to the paper is a spiral (also called helix). A load of 2.16N (sample holder and sample used) was used with a sliding speed of 96.1cm/sec along the spiral. Three samples were tested for each test condition (see Tables 3.1 and 3.2). In addition to samples previously exposed to thermal shock a hardmetal not subjected to thermal shock was also worn for comparison purposes. Faces on the samples which were previous polished were the once exposed to abrasive wear. One SiC paper was used for each sample tested i.e. until 180s. The duration of each wear test was 180s but at intervals of ±7 seconds the test was interrupted and the mass of the sample recorded. This allowed cumulative mass loss per time graphs to be plotted using Microsoft Excel.

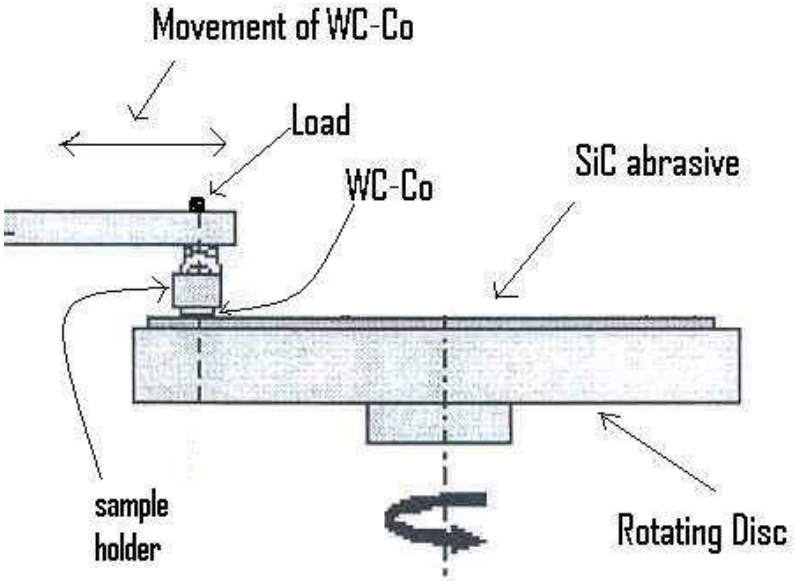

Figure 3.3: Schematic of 2-body sliding wear apparatus used for abrasive wear tests.

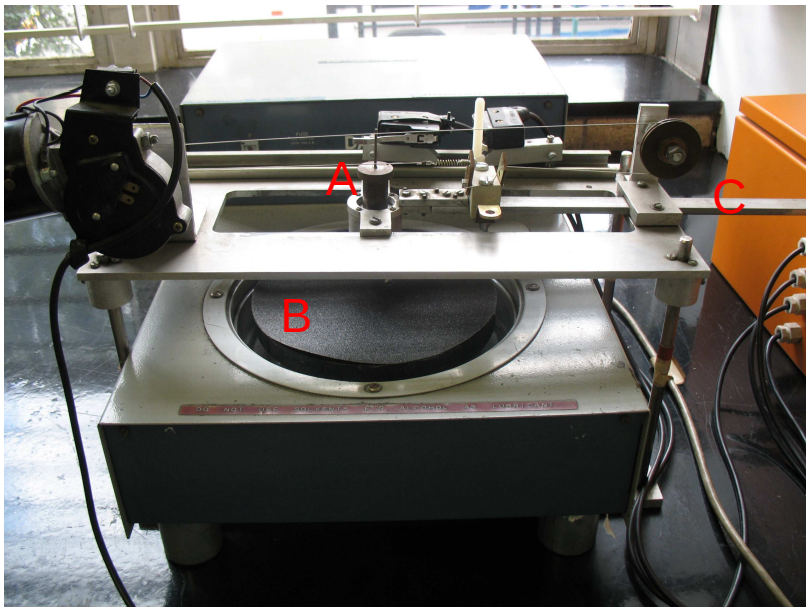

Figure 3.4: Photograph of the 2-body sliding wear apparatus, A is the sample holder, B is the SiC abrasive paper, C is the loading arm.

3.5 Microscopic Analysis

This section describes the different microscopes that were used for the project.

3.5.1 Stereo microscope

A Zeiss Stemi DV4 stereomicroscope was used in this project. This is a low magnification microscope which gives a 3-dimensional image of a surface. However, the micrographs do not retain the 3-d effect because the camera is mounted on only one ocular. This instrument was used to compare colour changes or damage features on the surface due to the different thermal shock conditions.

3.5.2 Optical microscope

A Carl Zeiss Axiotech 25HD optical microscope was used in this project. This microscope is connected to a camera and a computer with software for surface analysis. This was used for low magnification images.

3.5.3 Scanning electron microscope

Two different scanning electron microscopes were used, namely; a JEOL JSM-840 SEM and a JEOL 5600 SEM. JEOL 5600 SEM was used because of its high

resolution. Images were acquired using the backscattered mode at 20keV, and the EDS was used to analyze the elements present after the tests. The JEOL 5600 SEM is equipped with energy dispersive spectrometry (EDS) and a Centaurus backscatter system. This enables semi-quantitative chemical analyses. It is equipped with EDS software that allows spot or area analyses and chemical mapping of samples. Below are examinations and analysis that were done specifically for this project:

- In plan and cross-sectional views analyses of crack formation and cobalt depletion after thermal shock;
- Comparison of the wear scars of specimen subjected to various thermal shock treatments;
- Analysis of elements present after thermal shock.

3.5.4 Microprobe analysis

A Jeol JXA-733 Super-probe equipped with 4 wavelength dispersive spectrometers (WDS) and Noran Instruments energy dispersive detectors (EDS) with a NORVAR window was used for the microprobe analysis. This micro-probe EDS is capable of detecting all elements from boron upwards in atomic number.

The data was collected using a Noran Vantage Microanalysis System using backscattered electron images and line scan profile analysis. The conditions used were an accelerating voltage of 15kV and a beam current of 20nA.

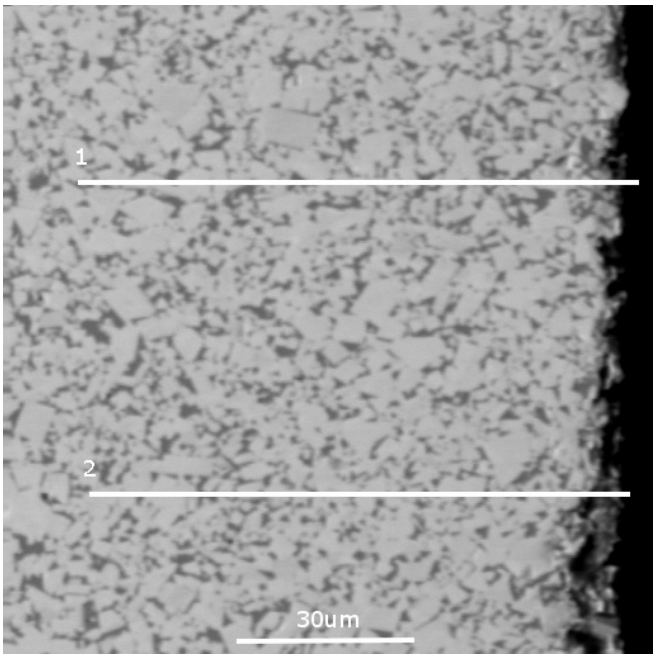

Figure 3.5: Micrograph showing two lines along which the sample was scanned from the core towards the edge.

On each sample the analysis was conducted at 50 points along each of the two lines similar to those shown in Figure 3.5. The elements Tungsten (W), Cobalt (Co), Oxygen (O) and Carbon (C) were line-scanned 50 points from the edge of cross-sectioned samples to the edge of the material. W, Co and C were scanned to detect any depletion as a result of thermal shock; O was scanned to detect any oxidation that may accelerate wear.

The data that is found from these analyses gives the relative concentration of tungsten, oxygen and cobalt at each of the 50 point along those 2 lines in weight percent. The weight percent was then converted into atomic percent using the following procedure: Atomic masses indicate how massive one atom is compared to another, which gives good comparisons of elements.

Consider a metal with four elements, A, B, C and D.

Then, $at\%A = \frac{n'A}{n'A + n''B + n'''C + n''''D}$

Where $n'A = \frac{wt\%A}{molarmassA}, n''B = \frac{wt\%B}{molarmassB}, n'''C = \frac{wt\%C}{molarmassC}$ and $n''''D = \frac{wt\%D}{molarmassD}$ [37]

The molar mass of Tungsten (W) = 183.85 g/mol, Cobalt (Co) = 58.933 g/mol, Oxygen (O) = 15.9994 g/mol and Carbon (C) = 12.011 g/mol.