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Use of FIB/SEM to assess the tribo-corrosion of WC/Co hardmetals in model single point abrasion experiments

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ABSTRACT

This paper describes the results of experiments where single point model abrasion experiments were carried out under a range of conditions of applied load and different media. FIB/SEM revealed that in air, damage occurred by binder extrusion in conjunction with fragmentation and fracture of the tungsten carbide grains, with re-embedment of fragments of carbide in the binder phase to form surface layers of mechanically mixed material. In HCl the binder was preferentially removed leaving the WC grains unsupported leading to further fracture and breakdown of the surface of the hardmetal. FIB revealed subsurface cobalt dissolution. A cobalt–nickel binder material proved the most versatile in resisting surface degradation in both ambient air and in HCl.

1. Introduction

Tungsten carbide based hardmetals, commonly featuring a cobalt binder, have been the preferred material choice in arduous applications covering a range of industries such as cutting tools, mining and oil/gas extraction to name but a few. Despite the genesis of the basic concept of a WC hard phase in a ductile metallic binder dating back to the 1920s [1], there are numerous components where a WC based material is still the preferred choice. They owe their longevity and versatility in no small part due to the utility of their hardness/toughness combination, which when optimised by microstructural refinement and a fitness-for-purpose approach, has enabled them, for the greater part, to adapt to increasingly stringent demands placed upon them in the aforementioned applications.

The response of hardmetals to abrasive or erosive conditions has been the subject of a number of studies over the last 30–40 years [2–5], in order to further the understanding of structure–wear relationships in these scenarios, and hence enabling a more informed material specification choice to be made for a particular application. Hardmetals have also been the subject of several studies performed under tribo-corrosive conditions; i.e. in which the conjoint action of wear and corrosion prevails [6,7]. These have been relatively few in number to date, possibly indicative of the more involved experimental conditions and ensuing analysis than pure abrasion or erosion.

Subjecting a hardmetal to a standardised abrasion or erosion test such as ASTM B611 [8] is almost trivial in nature in terms of test procedure and the results can be readily used in material ranking for comparison with other conventionally used mechanical properties such as hardness (e.g. Vickers, Rockwell etc) and fracture toughness (commonly the indentation method due to Palmqvist [9]). However, relating an individual abrasion or erosion event to the hardmetal structure local to the event is nigh impossible using a bulk wear test, though Gee et al. [10] have achieved this via eroding in a controlled manner; the so-called "stepwise" approach in which polished hardmetal surfaces are observed after being subjected to successive increments of erodent. The technique is useful in giving a certain amount of information on individual impact events, but is time consuming and retains the inherently random nature of a given impact event occurring in a bulk erosion test.

The response of WC/Co to the action of a loaded traversing indenter (commonly referred to as "scratch testing") has been the subject of a number of studies reported in the literature of late [11–14]. The technique can be regarded a model two-body abrasion scenario, with the indenter tip being a model abradant. The test gives the researcher far more control over surface degradation processes than a bulk abrasion test, as applied load, indenter geometry and traverse speed can be readily specified.

WC/Co is commonly found to undergo a number of degradation mechanisms as a result of scratch testing; both in the scratch itself, underneath and adjacent to it. Plastic deformation occurs in WC grains as evidenced by slip lines in scratch testing and also from subsequent EBSD (grain misorientation and zero indexing) [15]; WC is thought to deform by $\{10\bar{1}0\}$ prismatic slip [14]. Grain fracture, grain chipping, binder-rich transfer films, debris generation and re-embedment can also occur.

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In recent years, several important trends have been established with regard to the surface response of hardmetals to a traversing indenter [11–14]. Firstly, it is commonly found that higher loads promote greater incidence of slip in WC grains, increased occurrence of fracture, grain pluckout and communition. Repeat traverses also promote grain fracture; possibly indicative of a fatigue mechanism. Hardmetal composition also has a decisive role to play. In large ($4 > \mu m$) WC grains, more slip systems are active [16] and intergranular fracture occurs. Conversely, smaller WC grain sizes exhibit less slip and are more prone to pluckout. Higher binder volume fractions have a tendency to promote binder extrusion, communition, debris formation and re-embedment. Debris re-embedment is believed [11–13] to be a mechanism which mitigates further volume loss via binder extrusion; however its role in macroscale abrasion as seen in standardised laboratory tests is not clear, possibly due to the material removal rates being several orders of magnitude higher in the case of the latter.

Scratch testing is an established technique for assessing engineering coatings [17], the origins of which can be seen in the Mohs' hardness scale [18]. Scratching in engineering coatings, together with acoustic emission detection, is a well-documented and widely practised means of assessing coating adhesion [19,20]. In recent years there has been a growing interest in the friction of micro-scale contacts, the resultant degradation mechanisms and how both of these relate to macroscale behaviour.

2. Materials

Samples were provided by a number of hardmetal manufacturers under the auspices of the British Hardmetal Research Group (BHRG). Hardmetals subjected to scratch testing in the current study were previously characterised in terms of microstructure and mechanical properties; listed in Table 1. The inventory was chosen as it provides a convenient snapshot of the main microstructural variables in hardmetals; namely binder composition and grain size/binder intercept. Plane strain fracture toughness was generated from single edge notch beam specimens (SENB). WK (100) is the Palmqvist fracture toughness measured from cracks generated from a Vickers indentation produced by a 100 kgf load. This relatively high load was applied as lower loads of 30 and 50 kgf were insufficient to induce fracture from the indent corners in the mars11E grade; in general high loads are required to produce discrete and continuous Palmqvist cracks in hardmetals with coarse WC grain sizes and/or large binder intercepts; in the case of mars11E both these criteria are encountered.

Hardmetals as a generic material type feature a discrete hard phase (WC) in a metallic binder. As can be seen from Table 1 a range of hardmetal grades were used featuring differing WC grain sizes, binder contents and binder compositions. Binder compositions featuring nickel were chosen owing to their corrosion/abrasion–corrosion resistance relative to pure cobalt, which is seen in bulk abrasion scenarios [7]. Samples were supplied to NPL in a $40 \times 20 \times 5$ mm format. These were then cut to $20 \times 20 \times 5$ mm using a diamond cut-off wheel to facilitate hot mounting in bakelite and subsequent metallographic polishing to a 0.05 μm finish using colloidal silica, then annealed at 800 °C in vacuo to remove any surface residual stresses present.

Table 1
Hardmetal properties.

Grade	Binder (wt)	HV30	K_{IC}, MN m$^{-3/2}$	WK(100), MN m$^{-3/2}$	dWC, μm
shmcn5	4% Co, 1% Ni	1836	8.6	9.55	0.24
mars6ANI	6% Ni	1466	7.4	8.83	0.63
mars11E	11% Co	981	18.7	28.94	4.05
mars6A	6% Co	1583	9.7	10.73	0.68

3. Experimental procedure

The test system was designed (Fig. 1) to be used as either a bench top instrument or in an SEM. In the former configuration tests can be conducted in ambient air or controlled atmospheres. SEM operating capability meant adopting a new design capable of fulfilling the size constraints imposed by the SEM chamber dimensions, whilst retaining a relatively small working distance (6 mm) between the SEM polepiece and the sample surface.

The system features a flexure element designed [21] (Fig. 2) to support the probe, restrict probe motion to the two required orthogonal directions, generate the applied normal force, and to measure friction. Flexure element deflection is measured with capacitance displacement probes, where element deflection normal to the sample surface generates the applied load as the probe is brought into contact with the test sample; friction force being measured by monitoring deflection in the orthogonal direction. Probe position on the test surface is then adjusted by applying a feedback signal to a piezoelectric actuator mounted under the flexure to ensure that the applied load is constant. Position

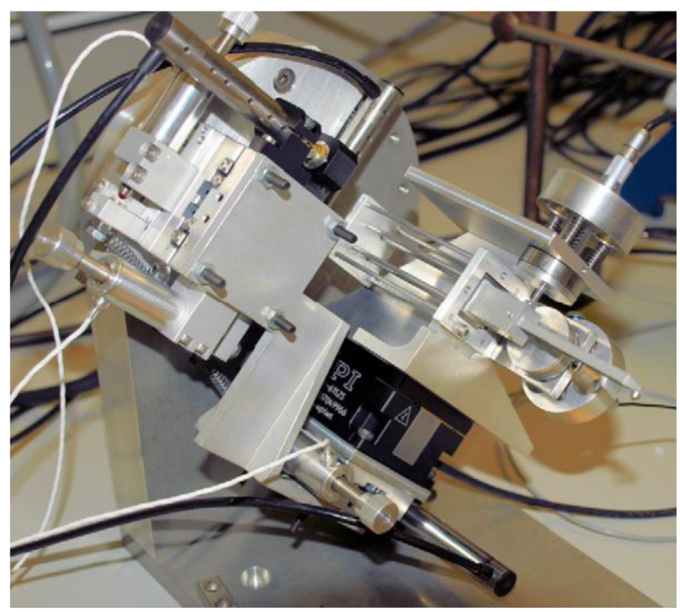

Fig. 1. Microtribometer developed at NPL.

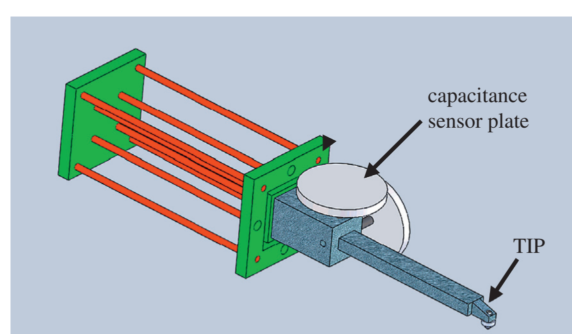

Fig. 2. Microtribometer-flexure element schematic.