

Micro-Ball Cratering of CVD Coatings

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1. Introduction

CVD coatings are being routinely used to improve substrate tribological properties for various applications such as cutting tools, valve components etc. The thickness of these coatings usually vary between 1-100 μm , thus evaluation of coating properties without the influence of the substrate is particularly difficult. Repeatable and well characterized methods are therefore required to evaluate tribological properties of these coatings. Micro-abrasion testing offers a simple, quick and reliable method to calculate coating and substrate wear resistance individually even for such thin coatings.

A hard spherical ball, normally either hard steel or tungsten carbide, is rotated against the sample in the presence of a slurry of fine abrasive particles, typically diamond or silicon carbide. The wear coefficient κ is calculated from wear scar volume, sliding distance and the normal applied load using the equation $V=\kappa SL$. The wear model assumes ideal spherical cap geometry with a same radius as the ball. However, the imposed geometry deviates from that of the ball owing to various interaction mechanisms, surface features and mechanical properties like hardness etc.

CVD coatings of Boron carbide, TiN-TiC, Diamond on various substrates have been tested in the present work and compared with virgin sintered substrates. Table 1 lists the various coated systems and materials tested. The effect of test parameters on wear rates has also been studied.

Specimen	Coating/Sustrate hardness, GPa	Thickness μm
TiN-TC/WC (4Ni,3.2Co, 0.8Cr)	20 \pm 2/ 14.5 \pm 1	8
B ₁₃ C ₂ /WC (6Co, 0.5Ta(Nb)C)	40-0/15.5 \pm 1	10
Diamond/W	60-75	15
WC (11Co)	14 \pm 1	
Sintered B ₁₃ C ₂	50-65	

Table 1. Properties of Materials tested

1.1. Nomenclature

- a Spherical misfit factor
- D_r Real crater diameter
- h Wear crater depth
- L Load
- L_c Load on coating
- L_s Load on substrate
- κ Specific Wear Rate
- κ_c Specific Wear Rate, coating
- R Ball radius
- S Sliding Distance
- t Coating thickness
- V Wear volume

2. Theoretical Basis

The Archard sliding wear equation can be shown to be applicable to abrasive wear of homogeneous bulk materials in micro-scale abrasion tests. However, inhomogeneous materials such as coated systems are not expected to have constant κ -values. At the onset of combined coating and substrate wear, the κ value is a weighted mean of the individual wear constants of the coating and the substrate (κ_c and κ_s , respectively). The wear equation in such cases can be reformulated as

$$dV/dS = \kappa L = \kappa_c L_c + \kappa_s L_s \quad (1)$$

Where, $\kappa = \kappa_c (\kappa_c, \kappa_s, L_c, L_s)$. The method used to calculate specific wear rates was developed by Kassman *et al*[1], to measure intrinsic abrasion resistance of thin coatings using a dimple grinder to grind craters into the surfaces of coating/substrate. The derivation considers ground spherical cap shaped craters as in Figure 1. Values of κ_c , κ_s and t are determined by fitting experimental crater depth vs. sliding distance values to the derived relationship.



Figure 1. Schematic of a wear crater.

It is readily shown that for $h \leq t$.

$$S = \pi h^2 (3r - h) / 3\kappa_c L \quad (2)$$

For $h > t$, the situation is slightly more complicated, and

$$S = \pi/L \left[\{2\pi(h - 0.5t) - th^2 + t^3h - t^3/3\} / \kappa_c \right] + \{t(h-t)^2 - (h-t)^3/3\} / \kappa_s \quad (3)$$

As $r \gg h, t$:

$$S = \begin{cases} \pi/L \cdot h^2 / \kappa_c & h \leq t \\ \pi/L \cdot [2t(h - 0.5t) / \kappa_c + (h-t)^2 / \kappa_s] & h > t \end{cases} \quad (4)$$

The Kassman method as modified by Gahlin *et al.* [2] takes into account the fact that the imposed wear scar deviated significantly from ideal spherical geometry. Due to the flattening of the crater bottom, the real crater depth is less than the apparent crater depth. Hence to calculate accurate wear volumes, a spherical misfit factor must be incorporated. The total volume can be shown to be

$$V_t = \pi/8R[(D_r - a)^4/8 + a(D_r - a)^3/3 + a^2(D_r - a)^3/4]$$

In the present work it has been assumed that the volume ratio of the crater (with respect to the total volume of the generated sphere) is constant

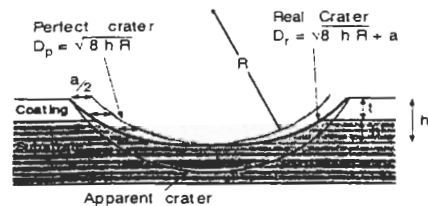


Figure 2 Deviation of real crater from perfect crater geometry [2]

3. Experimental Details

A Plint TE-66 micro-scale abrasion tester was used. The rig allows independent selection of the normal load, ball material (hard steel or tungsten carbide), rotational speed and sliding distance. The ball is held on a rotating shaft and the test surface is mounted on a counterbalanced beam, with applied normal load, that hangs vertically from a pivot.

The slurry used is 60µm diamond paste in distilled water. The optimum slurry concentration for near perfect wear scars was determined to be 0.5gm/ml at a drip rate of 45-60 drops/min. Slurry concentrations between 0.3-0.75gm/l have been used previously by various authors for tests on different materials with drip feed rates ranging between 15-150 drops/min. All specimens except the diamond coatings were tested

with a 25mm steel ball at rotational speeds of 75rpm and 150rpm with loads between 0.25-1 N. The diamond coatings were abraded against a tungsten carbide ball, 25mm diameter and identical rotational speeds and load. Initially, wear scar diameters were measured every 50 revolutions and after every 100 revolutions as the scar geometry becomes more uniform.

A 3-D talysurf 120L profilometer with surface mapping software and sphere fitting routines was used to analyze the wear craters produced. 3-D surface profilometry provides information on the real crater geometry such as actual depth, crater surface features and quantitative values for spherical misfit.

Pitted steel balls as suggested by Trezona *et al* [3] were found to have no perceivable effect on hard ceramic materials tested in the present work. The difference in measured crater diameter is less than 2%. Alsopp *et al.* [4] also found the deviation of aluminium crater diameters to be only 5%.

4. Results

Wear coefficients were calculated using eqn. 4. The applied normal load does not have a significant effect on the wear coefficient of any of the coatings or their substrates.

4.1 Coating Wear

Fig 3. compares the wear rate of the three CVD coatings with number of ball revolutions (sliding distance). The wear rates approach a steady state value as wear progresses, i.e. with increasing sliding distance. Low values of the wear coefficient in the initial stages of the test can be attributed to optical error in measurement as the scar is not well defined. The abrasion resistance increases with the coating material hardness. The wear coefficient for diamond is almost 15 times lower than the boron carbide coating.

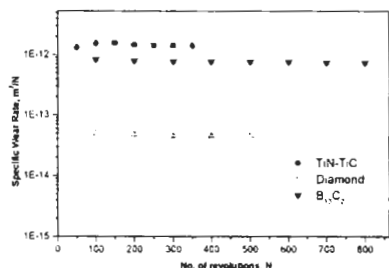
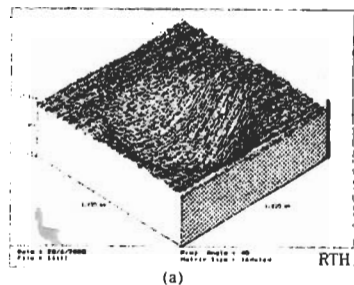


Figure 3. Comparison of Specific wear rate V_s No. of Revolutions of the three CVD coatings

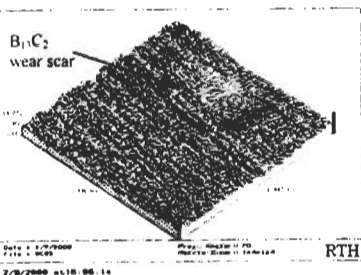
Figure 4 compares surface axonometric plots for TiN-TiC and $B_{13}C_2$ coatings. The geometry of the wear scar appear to be smoother in the case of the softer

TiN-TiC coating compared with the $B_{13}C_2$ and diamond coatings.

more uniform compared to the coated substrate wear scars as illustrated in Fig. 6.



(a)



(b)

Figure 4. Surface Axonometric plots of (a) TiN-TiC. (b) $B_{13}C_2$

4.2 Substrate Wear

The coating substrate wear rates are compared in Fig. 5 along with a virgin tungsten carbide (S11). The composition of the substrates are however not identical for all the coated samples (refer Table 1).

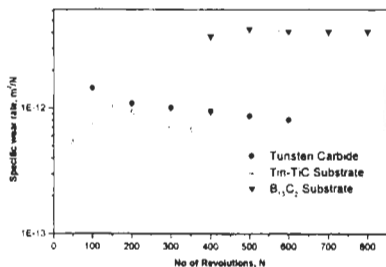
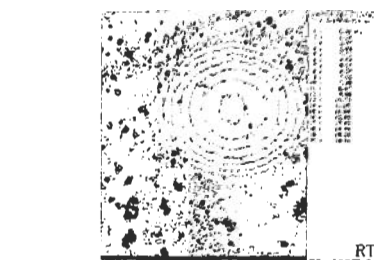
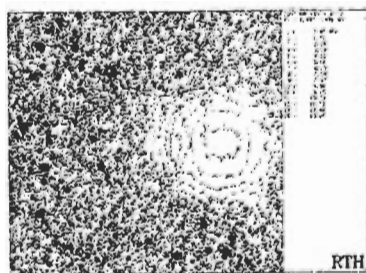


Figure 5. Substrate wear rates compared with virgin Tungsten carbide

The substrate wear rate is higher at the onset of abrasion and gradually decreases to a steady value as wear progresses. The wear coefficient of the substrates calculated from the compound wear equation are comparable to those of the virgin substrate. The wear scars on the virgin tungsten carbide are smoother and



(a)



(b)

Figure 6. Contour plot (a) Tungsten carbide. (b) Boron Carbide coated tungsten carbide

4.3 Effect of Ball rotation speed

Tests at various ball rotational speeds were performed on sintered B_4C and $B_{13}C_2/WC$ samples. For both materials the wear scars are smoother and more uniform at lower rotational speeds. The extent of three-body abrasion is also greater at low rotational speeds, and relatively less particle embedment and ridging is observed.

Fig 7 shows the wear coefficients for the two materials as a function of ball rotational speed.

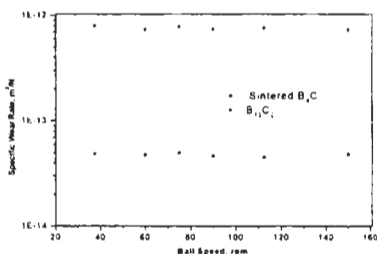


Figure 7. Specific wear rate vs Ball speed for $B_{13}C_2$ and sintered B_4C

4.2 Wear volume calculations

Equation 4 assumes that the wear crater assumes the imposed spherical geometry of the ball. The real crater diameter always deviates from the ball diameter. Also, in the case of a coated system, the sphere form of the coating wear scar differs from that of the substrate owing to the difference in their abrasion resistance. CASA, a surface assessment software provides the real crater diameter using the least squares sphere fitting algorithm on the crater surface profile obtained from the 3D talysurf.[5] The results obtained are more reliable for a homogeneous system as it's wear scar has a single sphere form as opposed to two sphere forms for a coating/substrate system.

The effective wear volume was also calculated using BODIES, a software which estimates the volume of a surface feature from the feature surface profile.

Table. 8 compares the wear volumes calculated by the three methods, i.e. experimental, corrected (using CASA) and real volume (using BODIES)

Volume, $\times 10^{-6} \text{m}^3$	Real	Sphere Fit	Experimental
TiN-TiC/WC	2.90	3.02	3.34
Sintered B_4C	1.22	1.65	0.98

Table 2. Comparison of wear volumes

5. Discussion

The surface topography of all the coatings as well as the virgin substrates appear to be smoother at the periphery of the wear scar. Wear tracks are observed more on the $\text{B}_{13}\text{C}_2/\text{WC}$ and the sintered B_4C scars. The gradual thinning of the coatings is also observed and contour maps obtained from 3D surface profiling show that wear scar generation become more uniform as wear progresses. The contour maps also suggest a layered development of the wear scars owing to wear of successive layers of the CVD coatings and sintered substrates. Smoothing of the scar with increasing sliding distance is also observed.

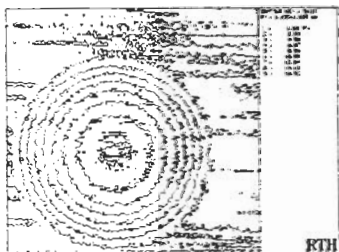


Figure 8. Contour map of TiN-TiC/WC. The wear scar appears to be symmetrical and the inner zones are more uniform in geometry

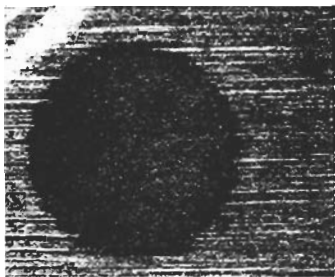


Figure 9. A typical sintered WC wear scar.(x100)

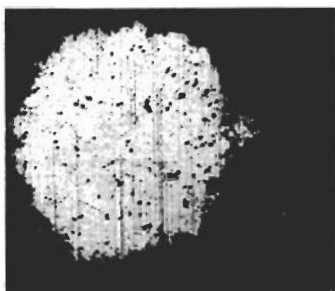


Figure 10. A sintered B_4C scar with visible wear tracks and ridging(x200)

The inhomogeneous microstructure of the B_{13}C_2 coatings owing to the presence of various phases result in certain zones experiencing greater wear, leading to a non-uniform geometry and topography.

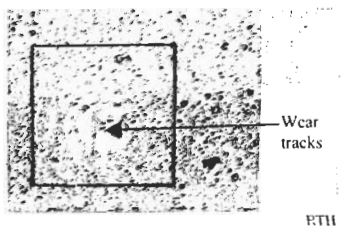


Figure 11. Contour map of a B_{13}C_2 scar. The scar appears relatively inhomogeneous

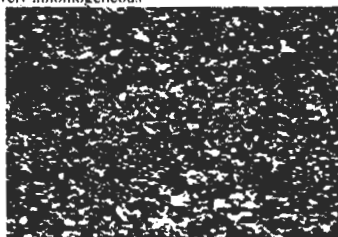


Figure 11. Surface structure of a sintered WC crater, no real interfacial cracking is observed, micro-chipping seems predominant (x1000)

Micro-chipping appears to be the predominant mechanism of wear compared to inter-crystalline fracture. Slurry particle embedment is seen in some wear scars mainly in the softer substrate zone. A Relatively low level of grooving in the direction of rotation suggest that the extent of abrasive particle entrapment is not very high Lower rotational speeds normally lead to the formation of uniform scar geometry and a smoother surface structure. A Relatively low level of grooving in the direction of rotation is observed for more cases suggesting that abrasive particles are uniformly entrained during the tests.

The sintered sample has a lower wear rate than the CVD structure. The extent of three body abrasion is greater on the harder materials.

6. Conclusions

- 1) Repeatable results were obtained for hard coated systems using the micro-abrasion rig.
- 2) The wear resistance of the coatings seem to be directly proportional to the hardness
- 3) No significant affect of load on the wear rate is observed.
- 4) Pitted balls do not affect any notable affect on neither wear rate nor scar geometry.
- 5) The calculated wear rates seem show good comparison with real volume loss calculations.

7. References

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