

Ball Cratering or
Micro-Abrasion
Wear Testing of
Coatings

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Abstract

This Measurement Good Practice Guide aims to provide introductory guidance on wear testing for coatings using the ball cratering or microabrasion wear test. Although the focus of the Guide is on abrasion testing, the use of ball cratering for unlubricated wear and friction testing is also covered.

The guide also contains a discussion on the most effective approach to wear testing and considers the different aspects that should be taken into account when designing a programme of friction and wear testing.

Specific guidance on how test conditions should be set and the procedures that should be used are given in Appendix A.

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1 Introduction

Friction and wear result from the interaction of contacting bodies that are in relative motion. They are a function of the detailed nature of the tribological system and the operating parameters.

Wear is a process in which material is removed from the surfaces of components, or by which these surfaces are seriously disturbed. There are a number of different types of wear and each one requires a different practical approach to wear testing.

A national survey in 1997 indicated that the cost of wear to UK industry was of the order of £650 million per year. Also for companies who have wear problems, the costs were typically about 0.25% of their turnover. In many cases these costs can be at least halved by making appropriate design and/or material changes.

Control of friction is also important. Often the goal is to reduce friction as much as possible to increase the efficiency of machinery. Furthermore, reductions in friction are often linked to reductions in wear so that as the friction is reduced, wear is also decreased.

This Measurement Good Practice Guide introduces the different wear mechanisms which occur in various types of industrial plant and machinery. The Guide then gives specific information on ball cratering or microabrasion tests for coatings, and gives recommended procedures for carrying them out. This Measurement Good Practice Guide is complementary to the handbook *A Guide on Wear Problems and Testing for Industry* [1], which contains information on the wear performance of different materials and reviews the different types of laboratory wear tests that are available for simulating practical wear conditions. It is published in parallel with a number of other Measurement Good Practice Guides on friction and wear testing on unlubricated sliding wear testing, abrasion testing of monolithic materials, and gas borne particulate erosion testing [2-4].

2 Types of Wear

2.1 Different Types of Wear

The seven main types of wear are shown diagrammatically in Figure 1, and are described in more detail in the following paragraphs:

Abrasive wear occurs when material is removed from the surface of a component by a cutting action or by a process of multiple indentation from abrasive particles (e.g. by rolling particles in 3-body abrasion). This may be an intended and controlled process in component manufacture, such as filing or grinding, or it may occur randomly in machine operation, such as the wear of digger teeth when working in gravel. It can also occur with two smooth surfaces rubbing together but with small hard contaminant particles getting in between them. Abraded surfaces show damage which can range from fine scratching to deep gouges. If the component is made from a ductile material, such as steel, the wear debris can be spiral in shape, similar to machining swarf. For very hard materials, the wear debris tends to be in the form of chips.

Adhesive wear is the surface damage and material removal which can occur when two smooth surfaces rub against each other. Such surfaces are never perfectly smooth and have high spots where the rubbing occurs. These local areas experience concentrated contact loads and interactions, and tend to adhere to each other dragging material away along the surface. This type of wear can occur in plain bearings and other interacting machine components, particularly if they are inadequately lubricated. The wear of brakes and clutches occurs by the same mechanism, and is kept under control by the use of dissimilar materials. Surfaces subject to adhesive wear can end up polished, with the generation of fine flakes of wear debris, or can show severe surface damage associated with surface dragging or even seizure.

Fretting is a particular form of adhesive wear that takes place when there are small oscillatory movements between two surfaces. These movements can arise from the deflection of machine components with clamped joints or press fits, or they may be intended small movements as in gear couplings. Fretting often produces fine powdered and oxidised wear debris around the components and there is usually surface damage and roughening of the joint surfaces. Fretting is also a limiting factor in the life of wire ropes. The individual steel wires rub against each other due to relative deflections when the load on the rope changes or when it is bent around a pulley. The oxidised wear debris often produces a red staining on the surface of the rope.

Erosion involves the removal of material from the surface of a component by the high speed impact of a liquid or of a stream of hard particles carried in a fluid flow. The two common types of erosion are cavitation erosion and particle erosion. **Cavitation erosion** occurs on components subject to low transient fluid pressures, such as ships propellers, and arises from the intense local impact of the collapse of low pressure vapour bubbles onto the component surface. **Particle erosion** occurs when a stream of hard particles is directed at a surface. This may be intended, as in shot blasting processes, or it may arise incidentally,

such as in pipe lines and associated components carrying slurries or crude oil containing sand.

Fatigue of surfaces can also lead to the loss of material when fatigue cracks in the surface join together to create loose particles. This surface fatigue can arise from a contact stress fatigue mechanism or from a thermal stress fatigue mechanism. Contact fatigue can occur in rolling contact where the passage of a ball or roller over a surface causes alternating tensile and compressive stresses, which can then create fatigue cracks. Thermal fatigue cracking can arise from the transient heating and cooling of a surface, particularly when combined with surface frictional forces such as in clutch plates and heavily loaded plain bearings. It can be particularly severe if a surface is in intermittent contact with a very hot material, such as molten metal.

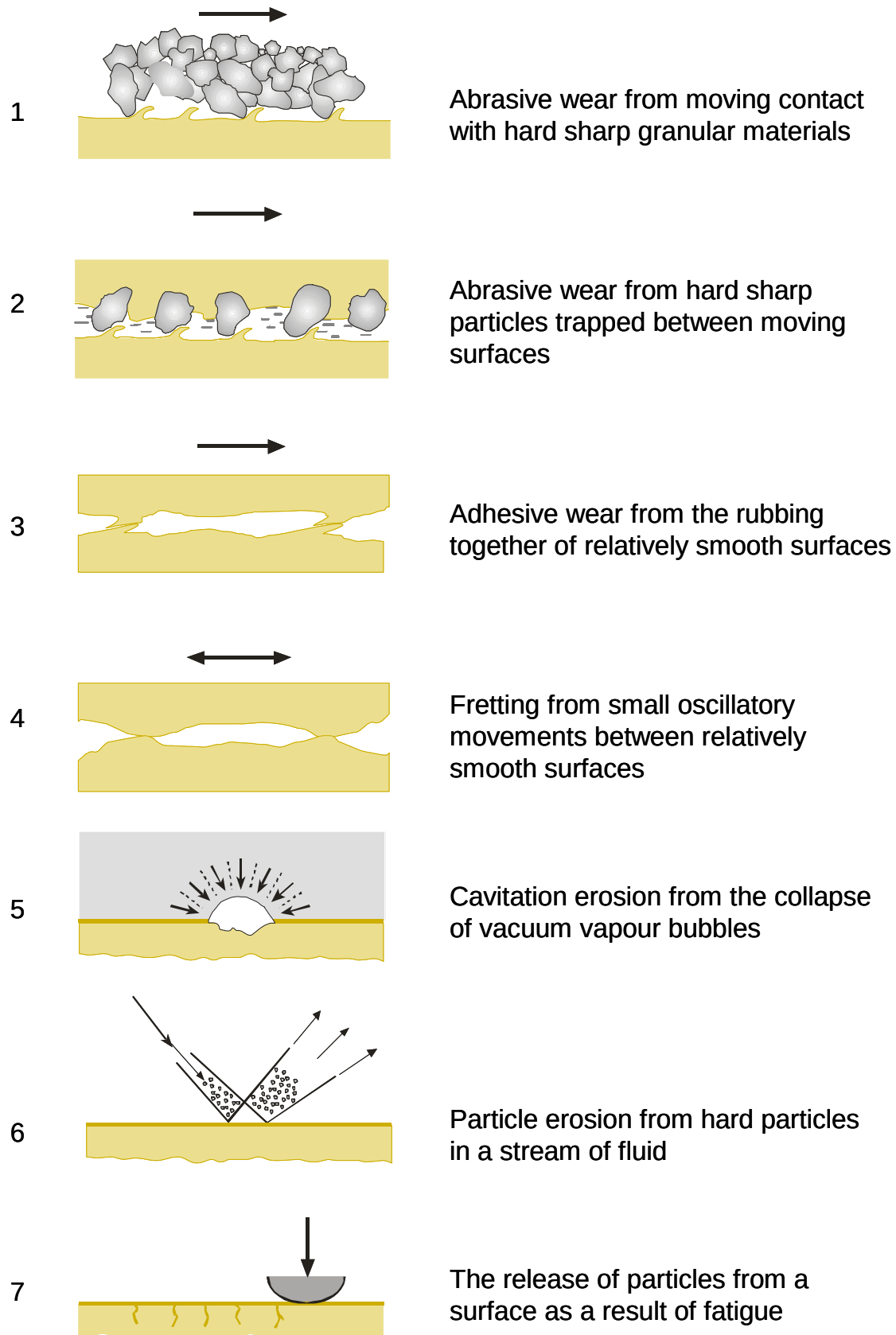


Figure 1, The types of wear that occur in industrial machines

3 Simulation of Wear with Laboratory Tests

It is important that in laboratory tests the conditions used should be appropriate and relevant to the real life conditions. It is generally not possible to completely simulate the conditions found in the application. This is because of the difficulty of defining the conditions that occur in applications, and also because of limitations in the test system that is being used to simulate the application, thus the match is unlikely to be perfect. Indeed, it is often desirable to simplify the conditions so that more control of the testing is obtained, leading to a better understanding of the results of the testing and their relationship to the test conditions.

When considering the design of a laboratory test and how well it simulates an application, it is useful to consider aspects of the different test parameters. These are:

- How important are the different parameters
- How controllable are the different parameters
- How measurable are the effects of the different parameters

In general, the importance of the various parameters will depend to some degree on the application that is being considered, but the most important parameter in any test must be the material structure and composition. Other parameters which have a major impact are load, speed, counterface material, abrasive material, abrasive shape, abrasive size, test-piece surface condition and the test environment.

Control of some test parameters is normally straight-forward, but there are some areas where this is much more difficult to achieve. In particular, in abrasion testing the results depend very critically on the shape of the abrasive particles, with many observers noting quite large changes in the magnitude of wear for different abrasives that had very similar shapes [5].

Measurement of many of the test parameters is also straight-forward but again there are some difficult areas, such as interface temperature measurement where practical methods do not exist for most materials.

It is important to ensure that the results from laboratory tests compare well with observed performance in practice. The final test for any product is obviously that it performs as expected in real life applications. The aim of laboratory testing must be to supply data on friction and wear behaviour that, if not completely equivalent, is relevant to the application. One way of ensuring this is to check that the mechanisms that are observed in the laboratory tests are the same as those observed in practice.

4 Background to Ball Cratering

One of the primary functions for engineering coatings such as TiN and diamond-like-carbon (DLC) is to improve the wear resistance and thus durability of components and products.

To determine these aspects of the performance of coatings, robust wear testing techniques are required.

Traditional techniques such as pin-on-disc or reciprocating sliding wear tests or the dry sand rubber wheel test have been used successfully [6,7], but, particularly for thin hard coatings, there can be considerable difficulties in performing tests [13]. This is often related to the fact that coating thickness constrains the volume or depth of material that can be removed before the coating is perforated. Thus for successful measurements of the wear of the coating, only small amounts of wear can be tolerated. Methods of measurement such as mass loss become ineffective, and even techniques such as profilometry often cannot be used for components with normal engineering finishes. This is because the depth of the wear damage is within the uncertainty of measurement caused by the original roughness of the surface.

One technique that has been considered for assessing the resistance of coatings to single or repeated abrasion events is scratch testing with single or multiple repeat scratches [9,10]. This shows considerable promise as a model test, but will not be considered further here.

This good practice guide describes ball cratering (otherwise known as micro-abrasion testing) which is a promising new technique for assessing the wear resistance of coated and surface engineered components. It was developed from two earlier techniques; dimpling to prepare transmission electron microscope samples ready for ion-beam thinning and cap-grinding for the measurement of coating thickness. In both cases spherical depressions were produced.

In ball cratering these techniques have been modified so that wear properties can be determined by performing tests where the test parameters are carefully controlled so that repeatable and reproducible measurements of the wear resistance of materials can be made.

Although the test was originally developed for abrasion testing of coatings, it is also possible to carry out unidirectional wear tests with the test system.

Currently there are no agreed test procedures in place for the use of this test. This is unfortunate as there are now a considerable number of systems installed both in Europe and worldwide (probably in the range 50-150). This lack of control is leading to a lack in reproducibility in results.

To rectify this situation, there are currently major efforts occurring in the USA through ASTM, and Europe through CEN to develop agreed procedures and standards for this test. In CEN, a draft standard is now being prepared for publication.

5 Overview of Test Systems

Three variants of the test system have emerged. In all the different types of system, wear is produced by pressing a rotating wheel or ball against the test sample and introducing an abrasive suspension into the wear interface (Figure 2). The three variants are described below.

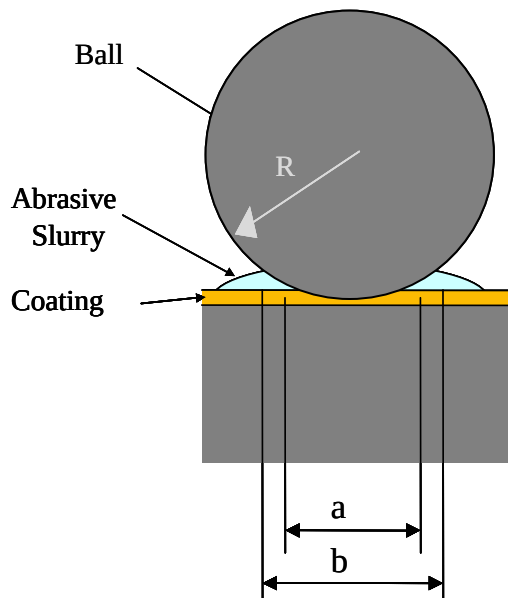
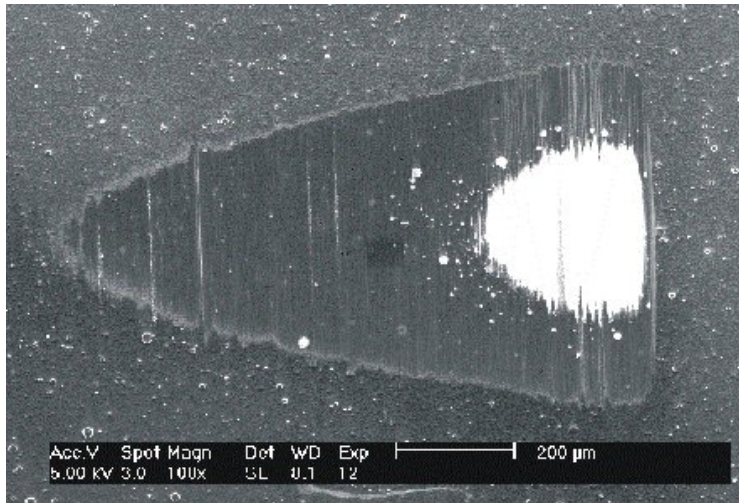


Figure 2 Principle of Test

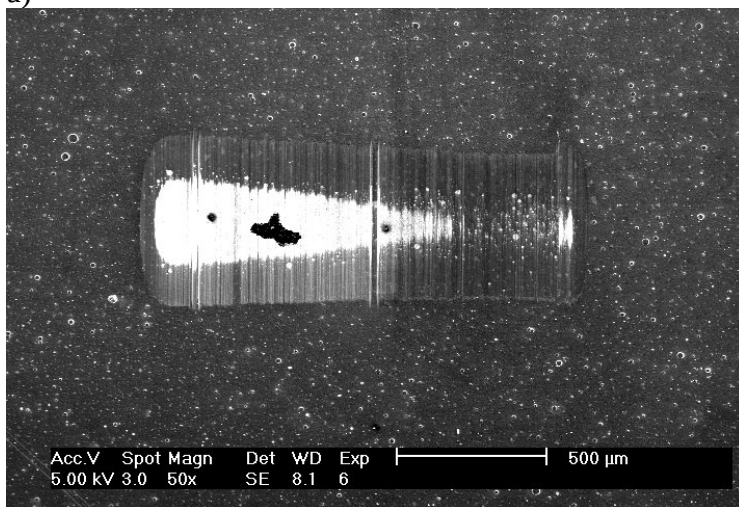
5.1 Rotating Wheel (Dimpler) Instruments

Here a spherical depression is produced by rotating the sample under the rotating wheel. To carry out tests effectively it is essential to accurately align the axis of sample rotation with the contact point between the test wheel and the sample. Although this has been achieved [11], the need for exact alignment makes this type of test difficult to carry out.

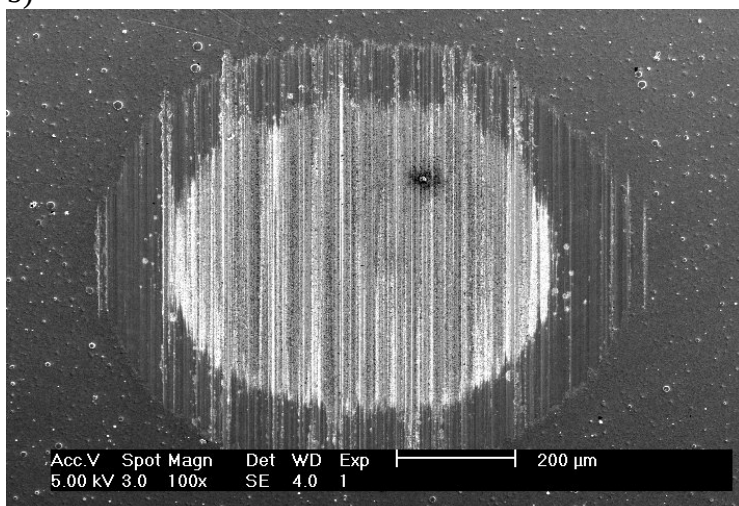
Tests can also be carried out without rotation of the sample. This ideally leads to a wear scar that has a cylindrical form, but alignment of the sample against the wheel is sometimes difficult to achieve (Figure 3) [12]. Thus wedge shaped scars are produced which become less symmetrical as the mis-alignment increases. If the wheel is shaped with a rounded edge or becomes worn, elliptical or even circular craters are created (Figure 3c).



a)



b)



c)

Figure 3 Experiments with rotating brass wheel pressed against non-rotating sample with 1 μm diamond paste, a) new wheel for duration of 30 minutes, b) new wheel for duration of 100 minutes, c) worn wheel for duration of 70 minutes

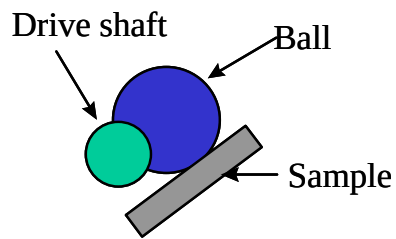
5.2 The cap grinding or free ball system

This test system (Figure 4a) uses a free ball which is driven by friction at the contact with a notched drive shaft. Because the ball is not directly driven, there is some uncertainty in the speed of the ball. However, the grip of the ball on the shaft can be improved by the use of rubber driving elements on the shaft [13]. The normal load is generated from the weight of the ball resting on the test sample. It is varied by altering the angle of the sample holding plate. However, it has been found that as the angle is reduced to increase the normal load, there is an increasing tendency for the ball to slide up the sample giving non-spherical craters [13]. There is also a potential source of error in the normal load due to the contribution to the effect of friction between the sample and ball which alters the effective weight of the ball. In the commercially marketed test system of this type [14], this potential error is reduced by using a load cell placed under the sample holding plate to measure the real normal load. Finally, it should be noted that for typical test balls, the maximum applied load that can be used is relatively small (about 0.4 N).

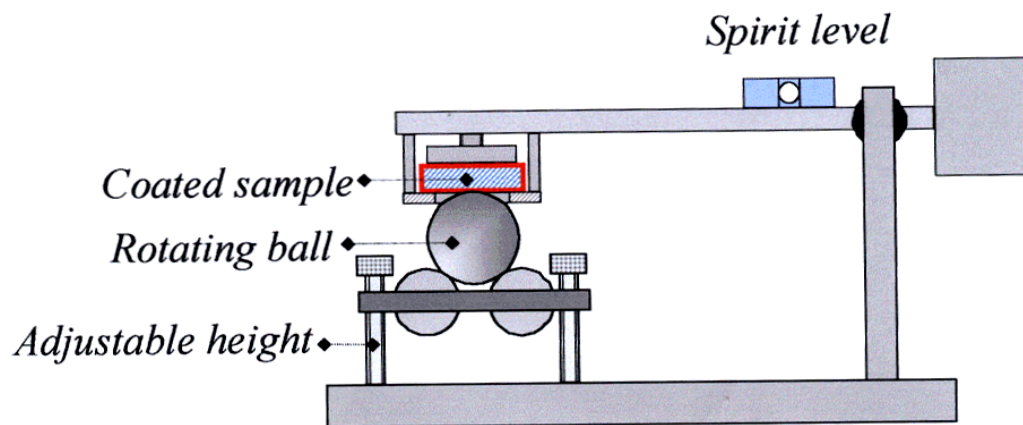
There is also another type of free ball machine which uses a 30 mm ball supported on grooves on two rotating shafts (Figure 3b) [15, 16]. The sample is clamped into a pivoted arm with dead weight loading applied directly above the ball. The face of the sample is pressed against the top of the ball and the load is applied range is from 0.5 to 5 N. Again this test system variant suffers from uncertainty in the speed of the ball because of the lack of the direct drive. However, the twin shaft arrangement avoids the potential error in applied load due to friction between the ball and the test sample, and also enables higher test loads to be applied than with the inclined plane system.

5.3 Directly driven system (fixed ball)

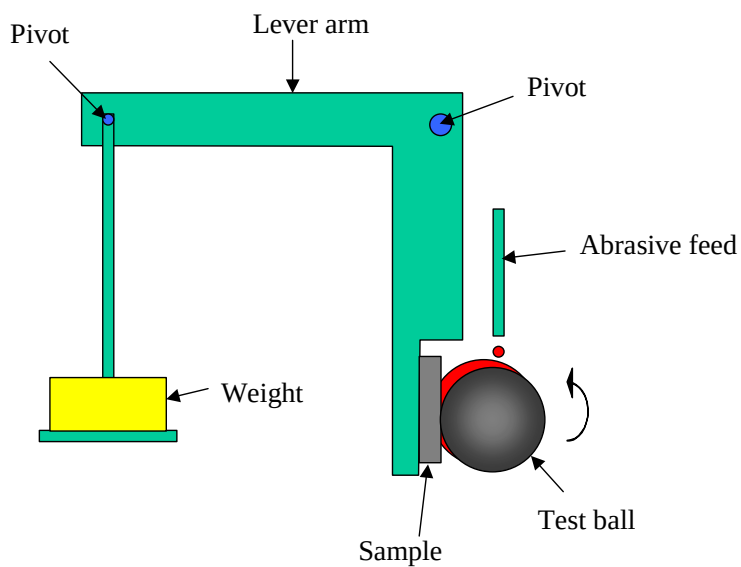
In this configuration (Figure 4c), the ball is driven directly by clamping the ball in a split drive shaft which allows balls to be removed and replaced easily. The sample is pressed into the rotating ball from the side by test loads placed on the weight pan. This design also allows for the application of loads exceeding the mass of the ball. Test systems of this type are now also marketed within Europe [17].



a)



b)



c)

Figure 4 Different test systems, a) free ball-single shaft, b) free ball-two shaft, c) fixed ball.

5.4 Relevance

There is considerable interest in these techniques for the abrasion testing of coatings. It is estimated that approximately 50 test systems of the different types described above have been set up in the last few years at a number of coating suppliers, users and research establishments. There are several reasons for this:

- The test equipment is relatively cheap to purchase or manufacture
- Test samples can be quite small as the size of the wear scar that is produced is small. This enables the technique to be used on small test coupons produced during the development of new coatings; as a technique suitable for quality control testing of coatings; and to perform tests on coated components to check the quality.
- It has the potential to be developed into an on-site test system, ie the test can be carried out in the field rather than the component having to come to the test laboratory.
- The test system seems simple and is thus attractive.
- The test system can also be used for thickness measurement.
- The test system can be used for profiling.

This latter point is particularly useful since wear scars often appear to be deep in systems with a hard coating on a soft substrate when in fact the actual loss of material is quite small with the depth of the wear scar caused by substrate deformation.

6 Basic Principles

Figure 1 shows the basic configuration of the ball tests. The ball is rotated against the test sample. Wear takes place to the sample and a crater is formed whose size provides a measure of the wear that has occurred. Often abrasive tests are performed by dripping abrasive slurry into the contact between the ball and the sample. Tests can be carried out limiting the duration of the test so that no perforation of the coating occurs. When perforation does occur, analysis of measurements of overall crater diameter (b in Figure 1) and the diameter of the crater in the substrate (a in Figure 1) for sets of tests performed to different test durations can yield wear rates for the substrate and the coating.

6.1 Tests Without Perforation

For monolithic materials, or for tests where perforation of the coating does not occur the volume V of a crater is given by

$$V = \frac{\pi b^4}{64R} \quad (1)$$

with b the crater diameter, and R the ball radius (for $b \ll R$).

This relationship assumes that the shape of the crater is conformal to the shape of the ball. This assumption has been shown to be true in many cases.

The Archard wear law states that the volume of wear $V = KSN$ where K is a constant (the wear rate), S is the sliding distance and N is the applied load. This has often been found to be true, but in some cases a strong dependence of wear rate on the number of revolutions and hence the sliding distance was found (Figure 5) [13].

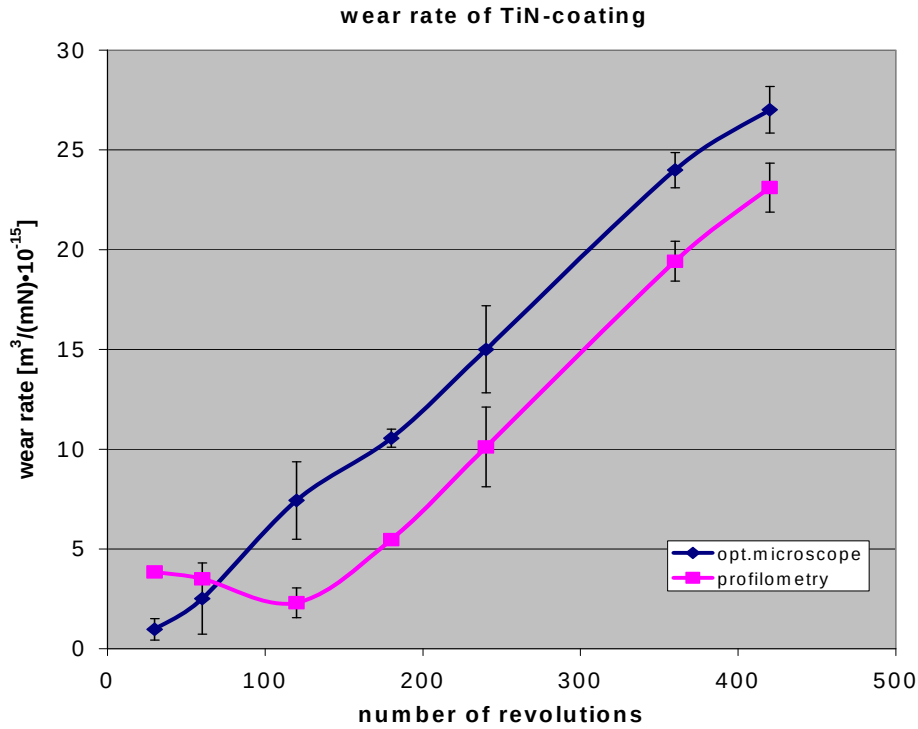


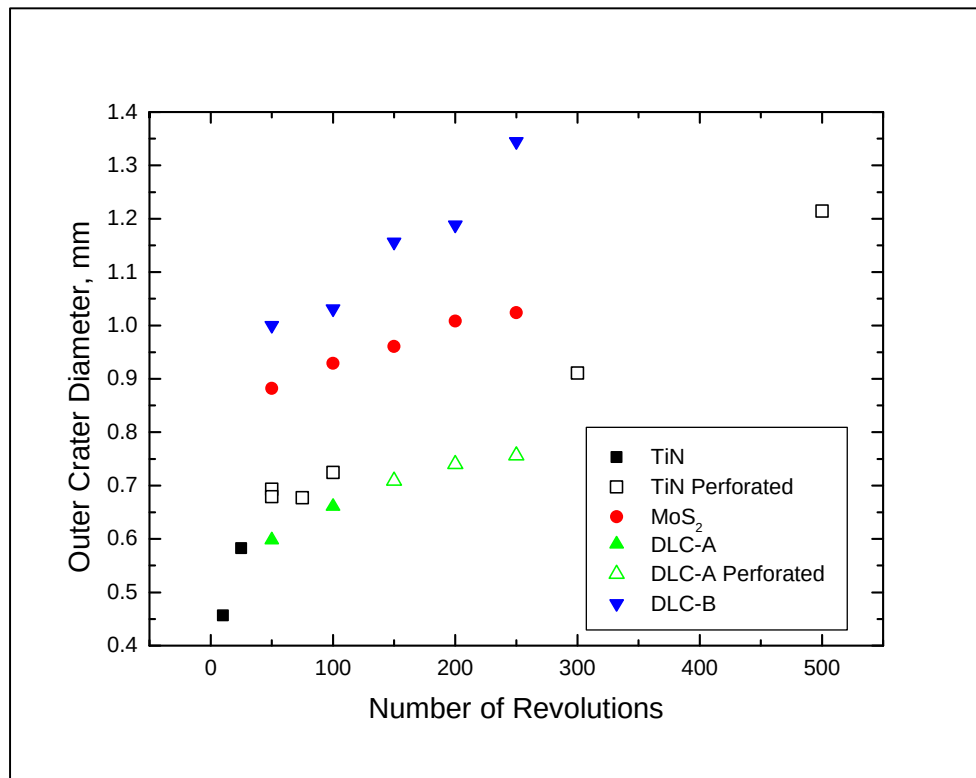
Figure 5 Wear rate of 3 μm thick TiN coating on steel. 1 μm alumina in glycerine slurry, 30 mm steel ball, 0.43 N load, 57 % RH, free ball-single shaft system [13].

However if the Archard wear law is followed, the wear rate K is described by

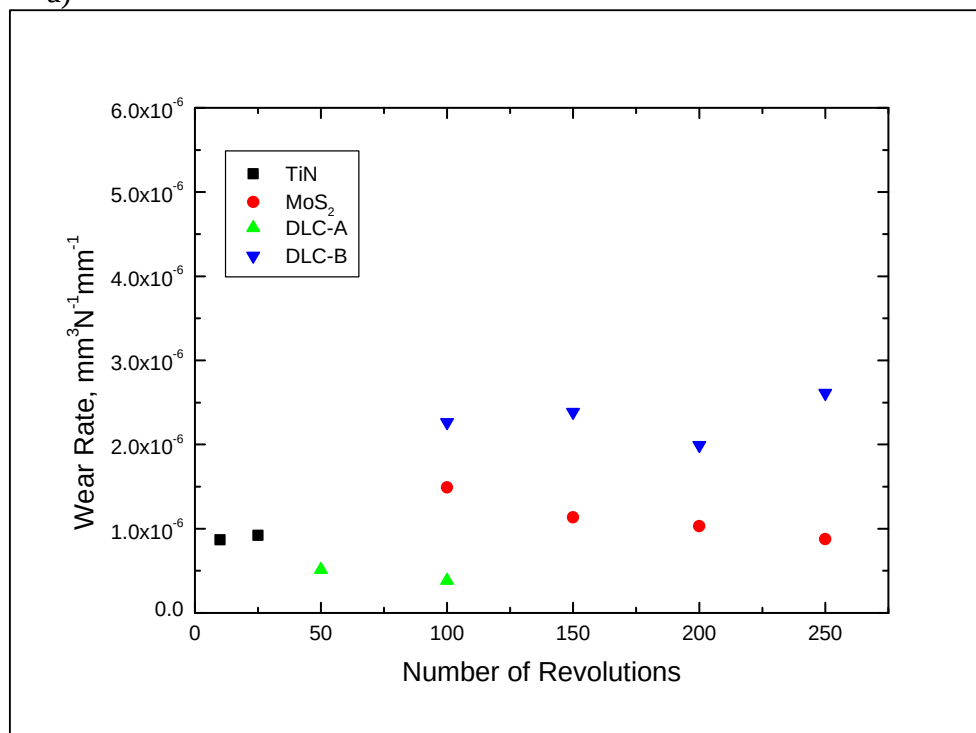
$$K = \frac{\pi b^4}{64R} \cdot \frac{1}{SN} \quad (2)$$

By performing tests without perforation of the coating wear rates can be measured easily for different coatings. This process is illustrated in Figure 6 where a number of experiments have been performed on a range of coatings. In Figure 6a, measurements of the overall crater diameter are given. As the test duration (number of revolutions) increases, the size of the crater increases. Perforation occurs for some of the coatings.

Equation 1 is used to calculate the wear rates shown in Figure 6b. Only the values for crater diameter for the craters where perforation does not occur are used. It can be seen that reasonably consistent (independent of sliding distance) wear rates are obtained for the four different coatings.



a)



b)

Figure 6 Tests performed on range of coated samples, a) increase in overall crater diameter with increased test duration, b) calculated wear rates from non-perforated craters.

Another example is shown in Figure 7 which shows the variation in wear rate with metal content for W-C:H and Ti-C:H coatings [13].

This technique has been shown to be effective for coatings as thin as 1 μm [18].

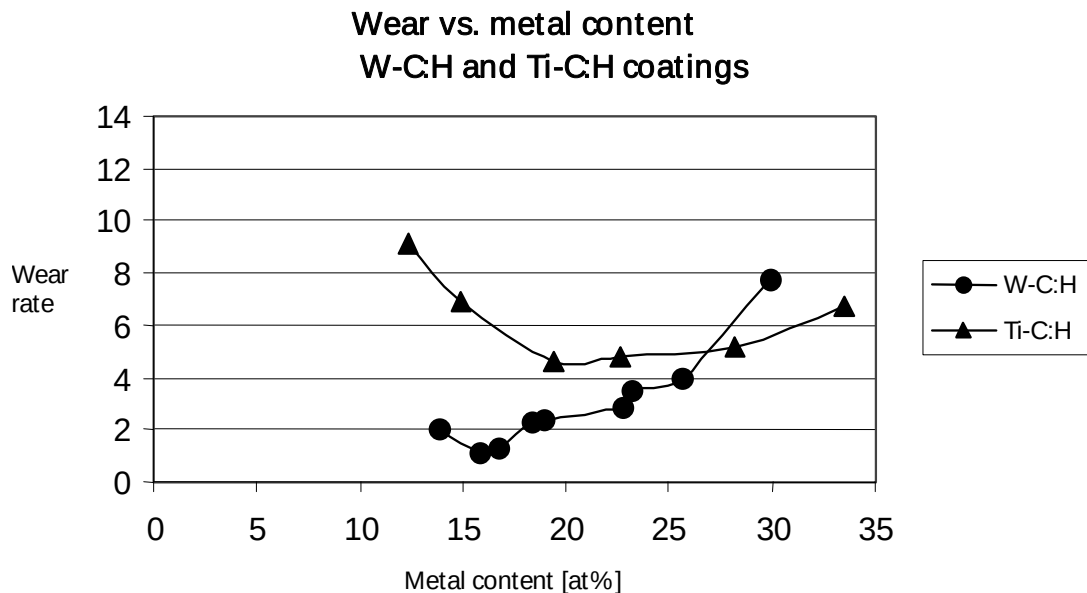


Figure 7 Use of non-perforating test to determine wear rate of metal containing DLC coatings as a function of metal content. Free ball-single shaft system [13].

6.2 Tests with Perforation

When perforation of the coating occurs, the procedure that was originally developed by Rutherford and Hutchings [19,20] can be used.

A series of tests are performed with different durations (Figure 8). The inner crater diameters (a in Figure 1) and overall crater diameter (b) are both measured. Typical results are shown in Figure 9. It can be seen that there is a steady increase in the size of both diameters. Note that perforation of the coating occurs at a relatively early stage.

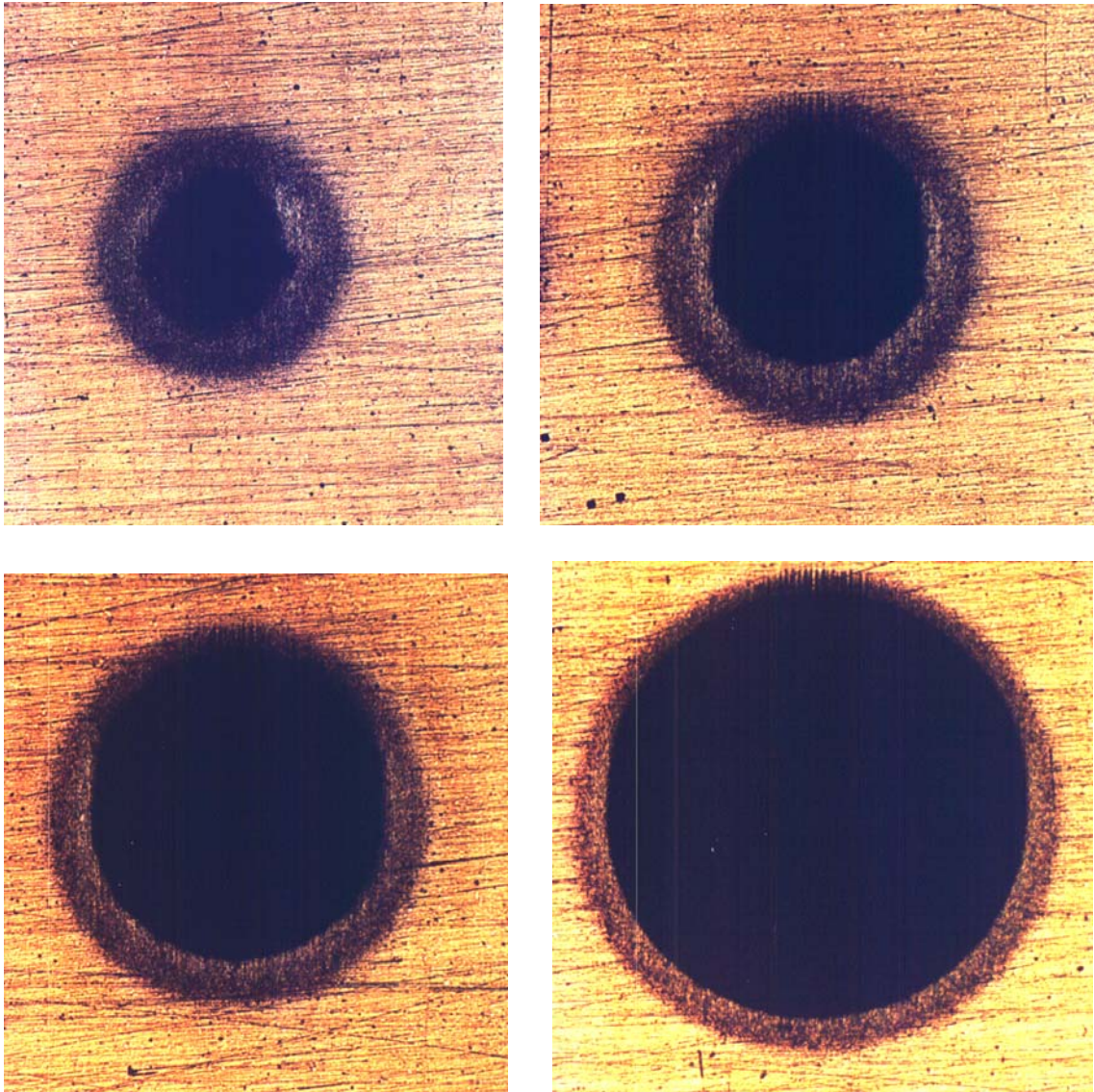


Figure 8 Craters produced by tests of increasing duration on TiN coated tool steel sample.

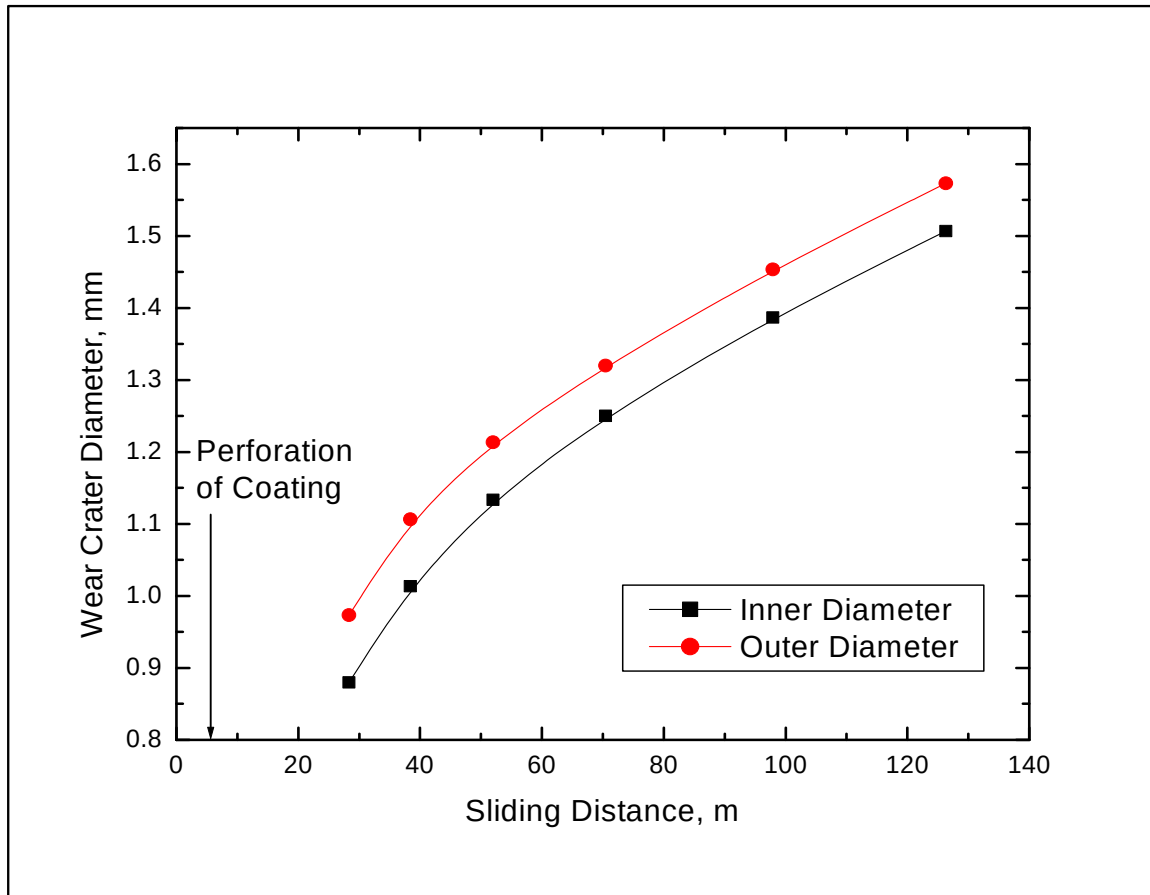


Figure 9 Measurements of crater diameter for series of tests on TiN coated steel sample. Rutherford & Hutchings [20].

The analysis assumes that progress of the wear in a cratering test follows Archard's law, but this needs to be modified to take account of the perforation of the coating after a short while. This is done by using an extended Archard's law for a coating-substrate system which can then be used:

$$SN = \frac{1}{K_c} V_c + \frac{1}{K_s} V_s \quad (3)$$

where K_c and K_s are the wear coefficients of the coating and substrate respectively, V_c and V_s the measured wear volumes and SN the sliding distance multiplied by the applied load.

There are different ways of implementing equation (3). It could be solved using a multiple least squares approach. The wear coefficients K_c and K_s are then considered both as unknowns. However, the equation is rather unstable, because of the relationship between V_c and V_s .

A better methodology is to rewrite the equation (3) as a linear function. Conventionally two forms are used. The first one is:

$$\frac{S N}{V_t} = \left(\frac{1}{K_c} - \frac{1}{K_s} \right) \frac{V_c}{V_t} + \frac{1}{K_s} \quad (4)$$

where V_t is the total worn volume. The formula is more stable when $\frac{V_c}{V_t} < 0.5$.

The use of formula (4) can be facilitated by using an estimation of the wear cap volume and by introducing the measured thickness t of the coating. This equation was derived by Rutherford and Hutchings [19]:

$$S N \left(\frac{64 R}{\pi b^4} \right) = \left(\frac{1}{K_c} - \frac{1}{K_s} \right) \left(\frac{16 R t}{b^2} - \frac{64 R^2 t^2}{b^4} \right) + \frac{1}{K_s} \quad (5)$$

The coating thickness, t , can be calculated from the same micro-abrasion test or can be measured separately (Annex B).

A second way of rewriting equation (3) is:

$$\frac{S N}{V_t} = \left(\frac{1}{K_s} - \frac{1}{K_c} \right) \frac{V_s}{V_t} + \frac{1}{K_c} \quad (6)$$

The practical version of this equation is (see Rutherford and Hutchings [19]):

$$S N \left(\frac{64 R}{\pi b^4} \right) = \left(\frac{1}{K_s} - \frac{1}{K_c} \right) \left(1 - \frac{16 R t}{b^2} + \frac{64 R^2 t^2}{b^4} \right) + \frac{1}{K_c} \quad (7)$$

An example of the application of the analysis is given in Figure 10 where equation 5 has been applied to the analysis of the results from Figure 8. In this case a very good linear fit has been achieved leading to confidence in the calculated values of the wear rates for the coating and the substrate.

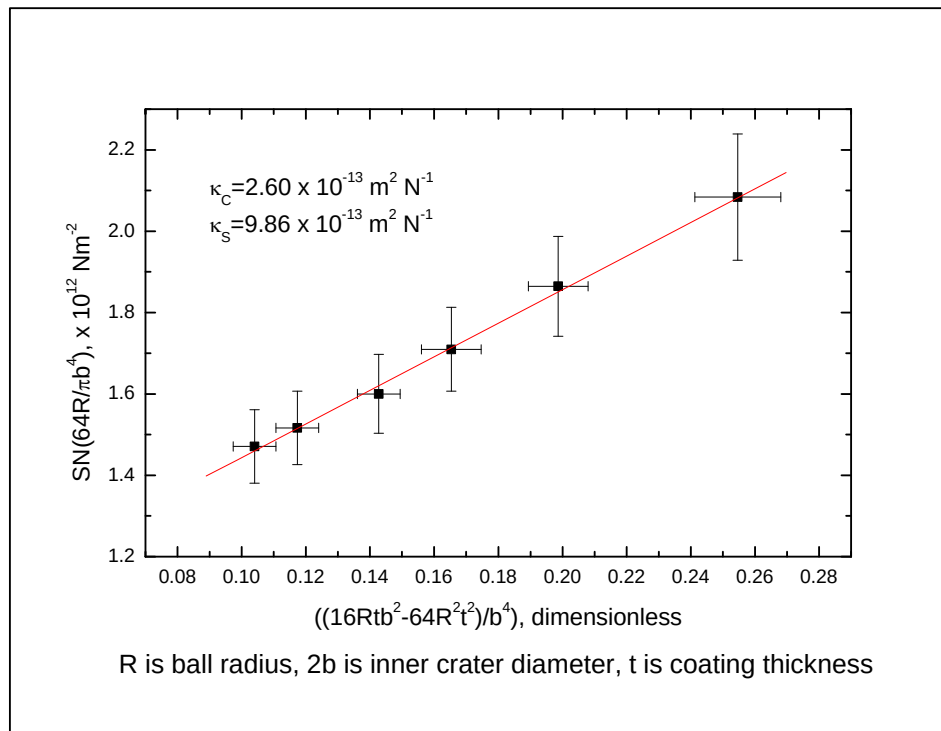


Figure 10 Analysis of ball cratering results of set of tests on TiN coated steel resulting in calculation of wear rates for steel and coating. From Rutherford & Hutchings [20].

7 Sliding Wear Testing

Recently the ball cratering test has been modified to give the capability for performing sliding wear tests (Figure 11) [21]. The test system has been modified by adding strain-gauged leaf springs to enable the measurement of frictional force generated at the contact. A displacement transducer is also fitted to measure the movement of the sample towards the ball as the sample wears.

The wear scars formed in a test between a steel ball and TiN coated tool steel sample are shown in Figure 12. A large volume of brown debris was formed on the coated sample. The brown colour of the debris indicates that the debris is likely to be iron oxide. This is confirmed by the brown colour of the scar on the ball. When the scar is cleaned to remove debris, reduced wear at the centre of the scar becomes apparent[†] (Figure 13).

Typical friction and wear displacement results are shown in Figure 14. In this test the friction coefficient is high at about 1, and subject to periods of highly variable friction (shown by the high standard deviation in the friction values at certain points). The wear displacement steadily increases as the test proceeds until a final average displacement of about 4 μm was observed.

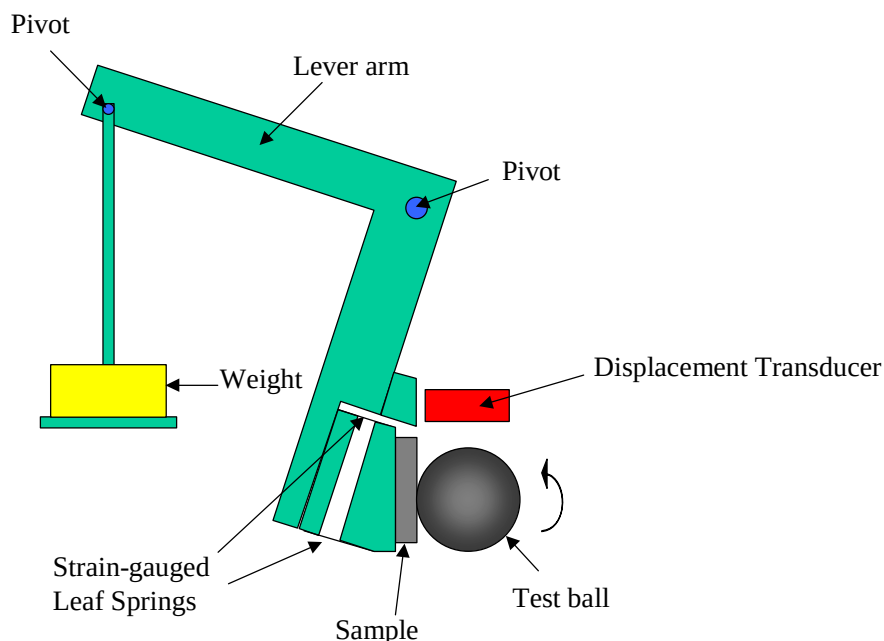


Figure 11 Modification of ball cratering tests to add friction measurement capability for sliding wear tests.

[†] see later section on load effects.



a)



b)

Figure 12 Wear scars from dry ball cratering sliding wear tests on TiN coated steel sample, a) crater on sample, b) track on ball.

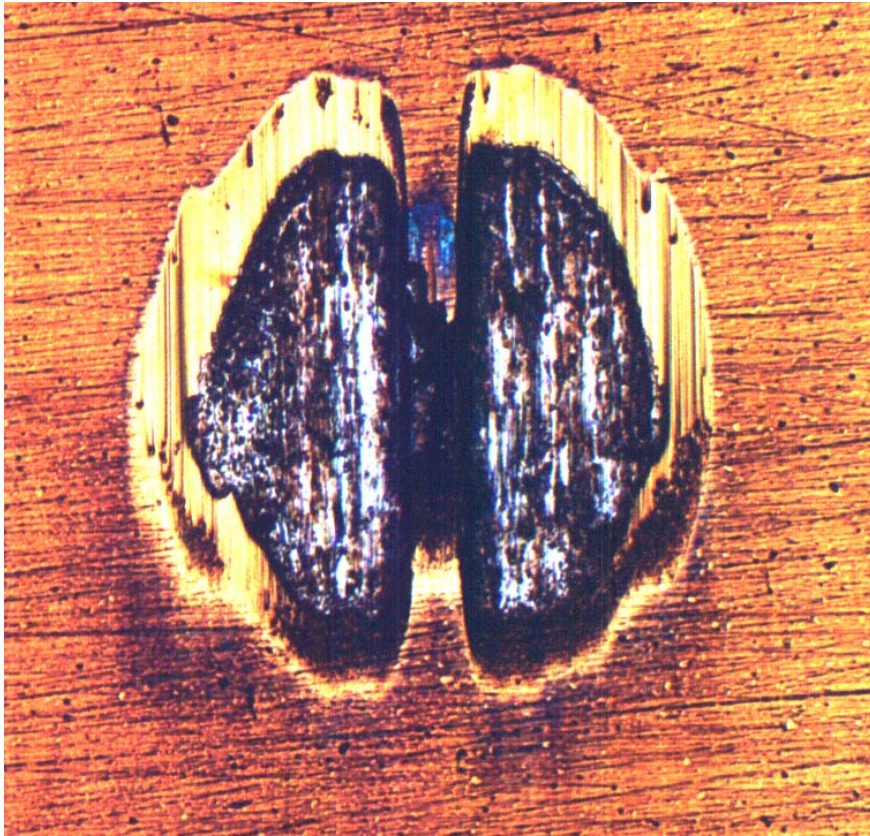
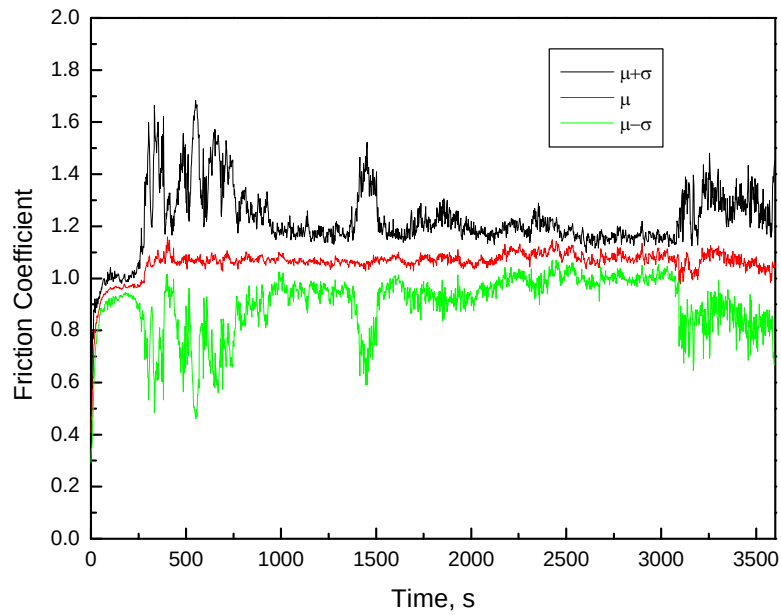
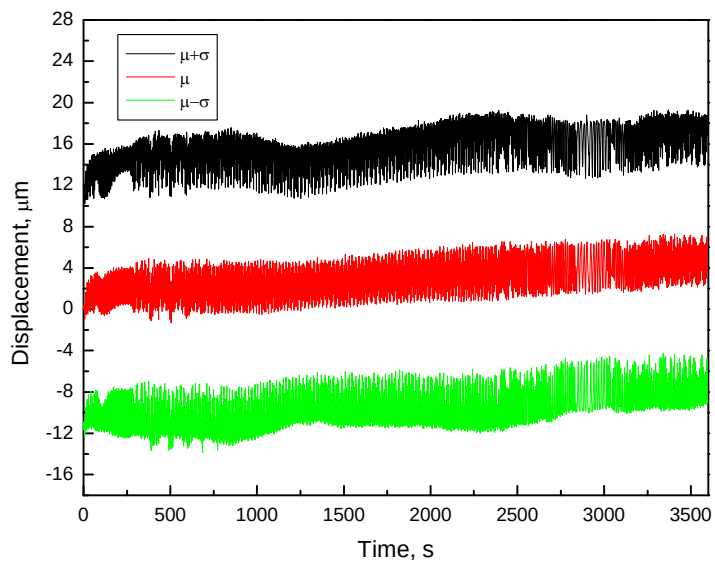


Figure 13 Cleaned wear scar from dry ball cratering sliding wear tests on TiN coated steel sample showing ridging.



a)



b)

Figure 14 Results of on-line measurements of friction coefficient and wear displacement for a test with a steel ball and TiAlN coated tool steel sample with an applied load of 4.7 N, a) friction coefficient, b) wear displacement.

8 Sectioning

Ball cratering can also be used for the more traditional roles of forming taper sections. This can be seen in Figure 15 which shows a crater through a multilayered coating. The structure of the coating is clearly shown at the edge of the crater.

If the ball crater test system is being used to carry out sectioning rather than to perform a wear test, it is important to optimise the test conditions so that a smooth crater surface is obtained. This is normally achieved by using low applied loads and fine abrasives.

Precise positioning can be achieved which enables the technique to be used as a method of measuring the profile of wear tracks from other types of wear tests such as pin-on-disc wear tests. This can give a very accurate measure of wear and also has the advantage that sub-surface deformation under the wear track can be detected. This is important when wear tests are carried out on wear resistant coatings, such as DLC, on softer substrates where an apparent wear track is formed which is shown to be due entirely to sub-surface deformation (Figure 16) [22].

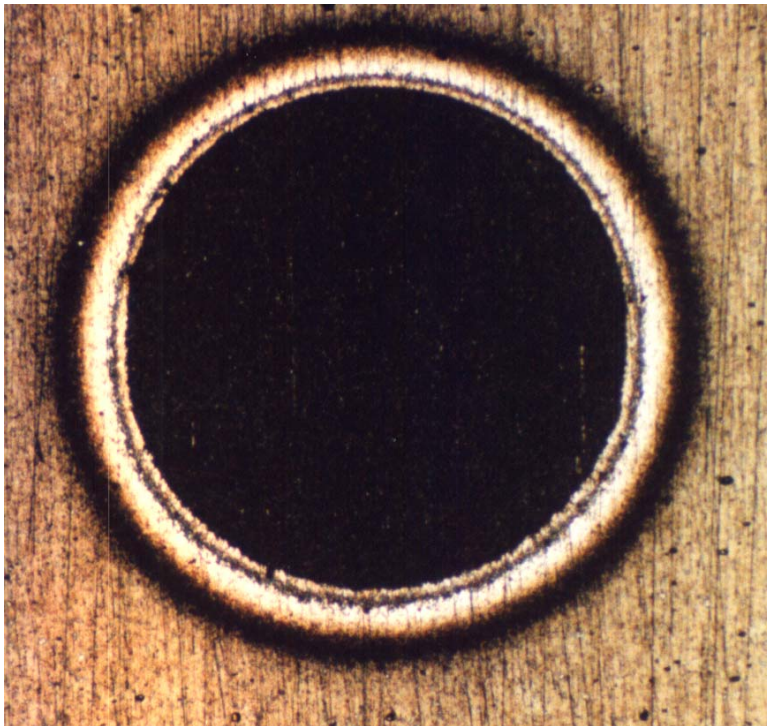
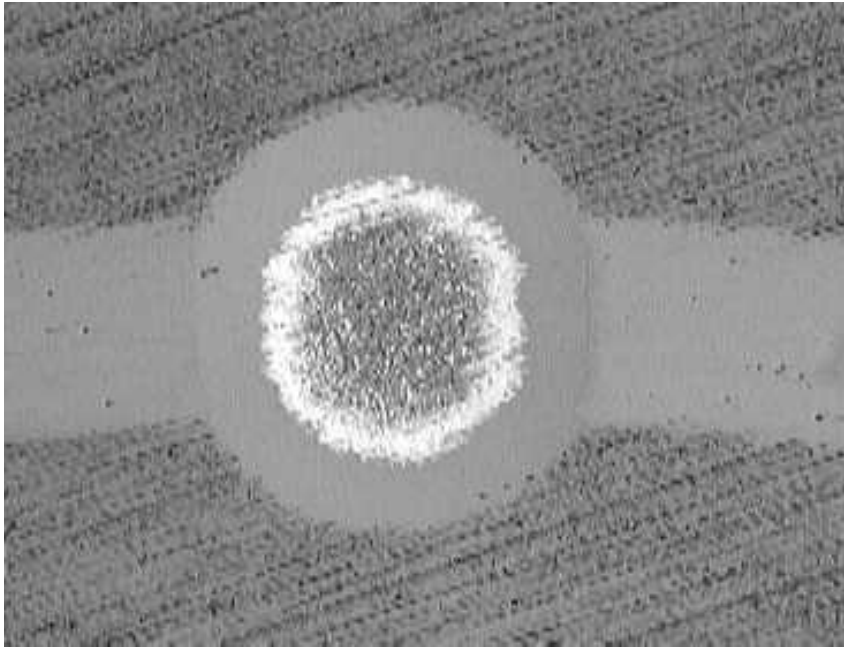
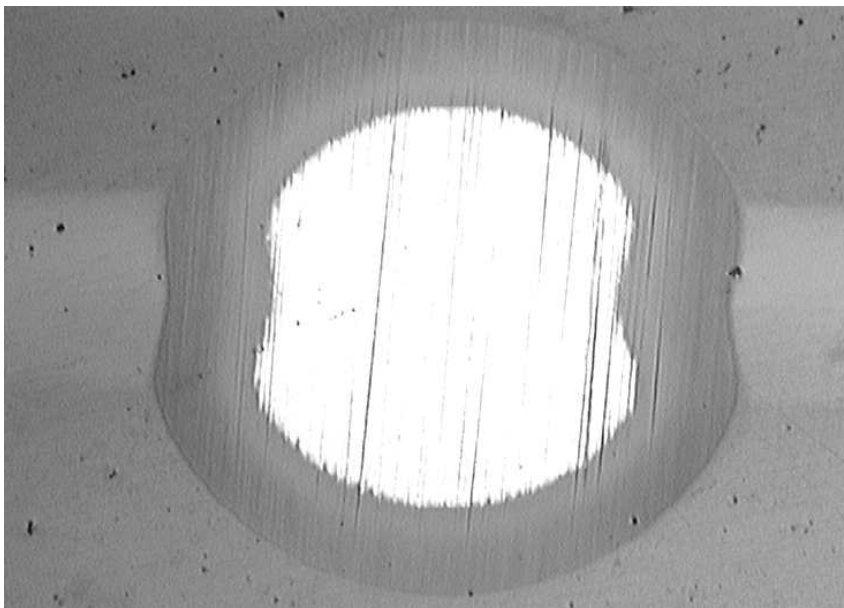


Figure 15 Ball crater showing how techniques can be used to investigate structure of coatings.



a)



b)

Figure 16 Examples of use of ball cratering for taper sectioning, a) pin-on-disc wear track, b) apparent wear track showing clear plastic deformation of substrate under track.

9 Parameters Affecting Results

There are a large number of parameters that affect the results of ball cratering tests. These include:

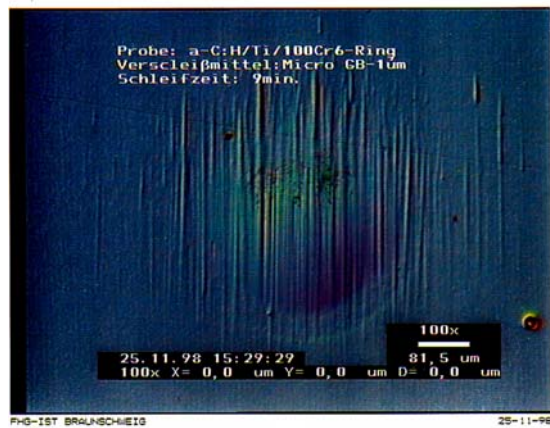
- the abrasive material
- the abrasive size
- the abrasive shape
- the abrasive loading
- the type of suspension fluid
- the load
- the speed
- the ball material
- the ball surface condition.

9.1 Abrasive

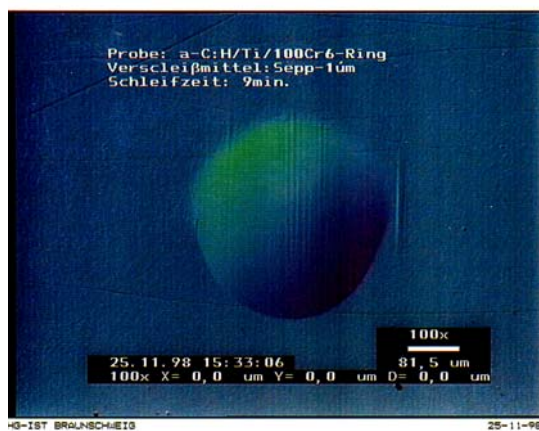
The abrasive material is clearly important. The main reason is the hardness of the abrasive, and in general, the harder the abrasive, the higher the wear rate. However, it is important to realize that it is the relative ratio of hardness between the abrasive, the ball material and the sample that is important.

The size of the abrasive is also important. It has been shown from consideration of the frictional forces that act between the test surfaces and the abrasive particles that there will be upper limit of abrasive which can be drawn into the interface between the ball and the sample [18]. Clearly if no abrasive is present at the interface, the wear mechanism changes radically. For the size of balls that are normally used (25 mm diameter), this limit will be in the range 10-20 μm diameter. This is the main reason that this test is most appropriate (for abrasion testing) for use with fine abrasives. Typically abrasives less than about 5 μm diameter are used.

Under some conditions, craters made with coarse abrasives can be difficult to measure as the edge of the craters are obscured by grooving from the abrasive. When this occurs, the use of finer abrasive can give better crater definition (Figure 17) [18].



a)



b)

Figure 17 Effect of abrasive size on crater, a) deep grooving from coarse abrasive b) well defined crater from fine abrasive [13].

As with all abrasive tests, the shape of the abrasive is important. Abrasives with more angular shapes will give higher loading when they are pressed and moved against the sample. For this reason abrasive damage and wear are likely to be higher for more angular shaped abrasive particles.

9.2 Abrasive Loading and Load

The effect of applied load and the concentration of abrasive in the slurry are linked. As the load is increased there is a transition from a three body rolling wear mechanism to a two body grooving mechanism. Conversely, as the volume fraction of abrasive is increased, there is transition from two-body grooving to three-body rolling. These transitions can best be expressed [23] by a wear mechanism map (Figure 18). Typical micrographs of the surfaces of samples produced in these different regimes are given in Figure 19.

The gross appearance of craters tested in the two body grooving regime are typically shiny in appearance (Figures 19b and 20) with a regular array of grooves over the surface. By contrast, surfaces of samples tested in the rolling regime are dull in appearance (Figure 19a) with much more random damage.

The slurry concentration also affects the magnitude of wear that occurs, with higher wear in the rolling wear regime at higher slurry concentrations [23] (Figure 21).

When the load is increased further, the phenomena of ridging occurs (Figure 22). Little or no wear occurs at the centre of the scar because of blocking of abrasive ingress at the centre of the contact resulting from the high Hertzian contact pressures at the centre of the contact. This prevents abrasive from entering this contact region and thus reduces or eliminates abrasive wear in the centre of the scar. The ridging prevents further increases in wear with increasing load above a critical load value (Figure 23). As well as the phenomenon of ridging, there is evidence that at high loads abrasive can also be embedded into the sample surface leading to erroneous results (Figure 24) [13].

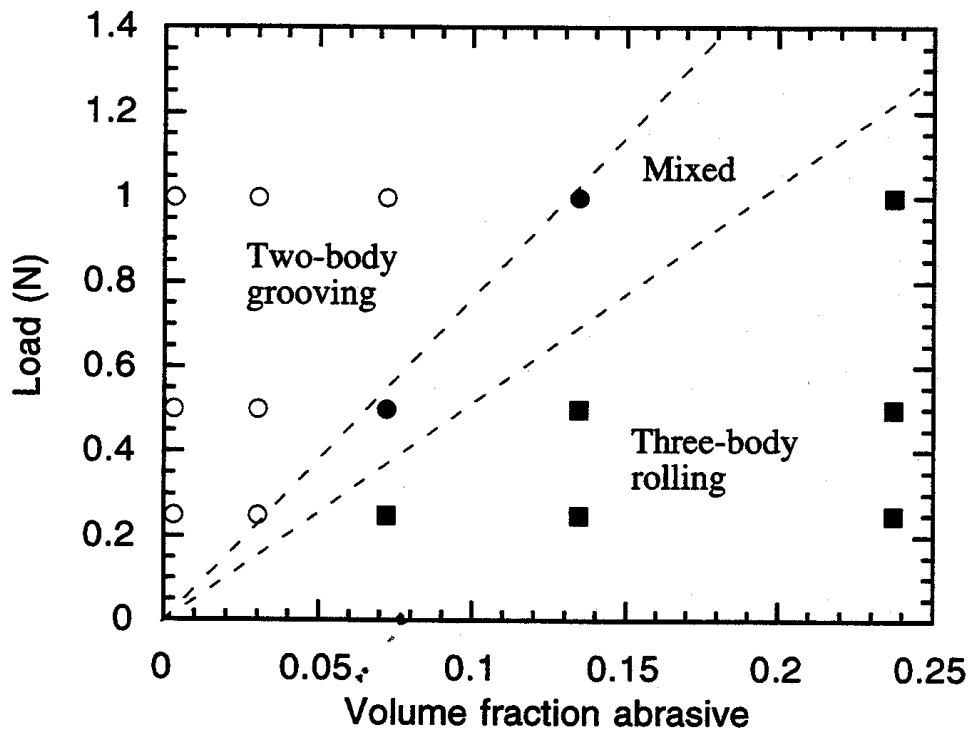
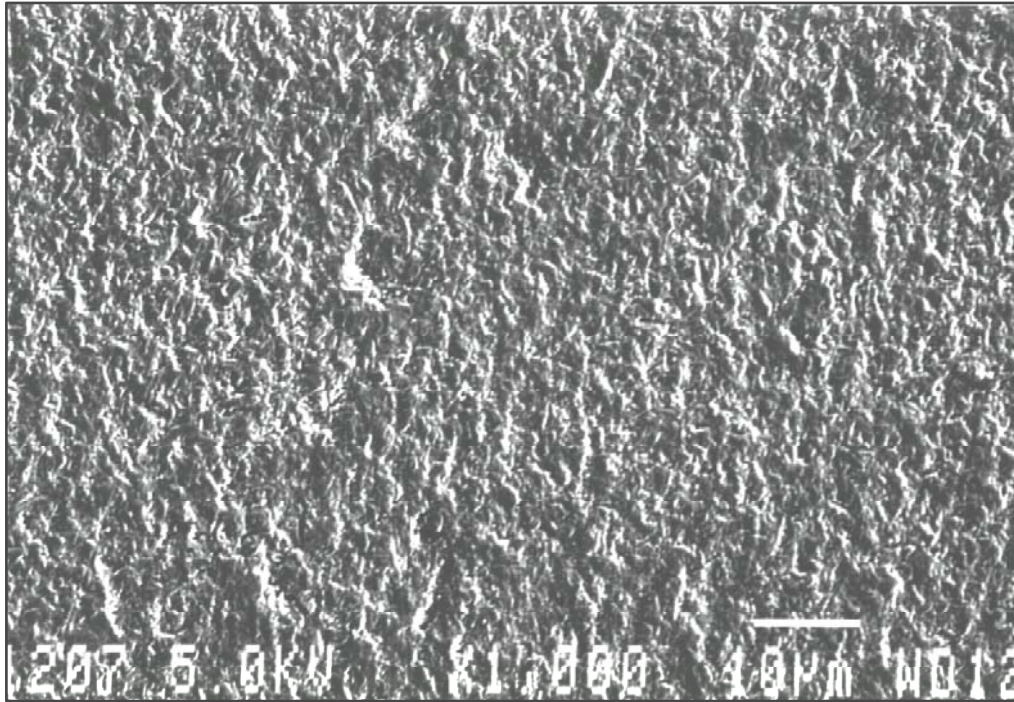
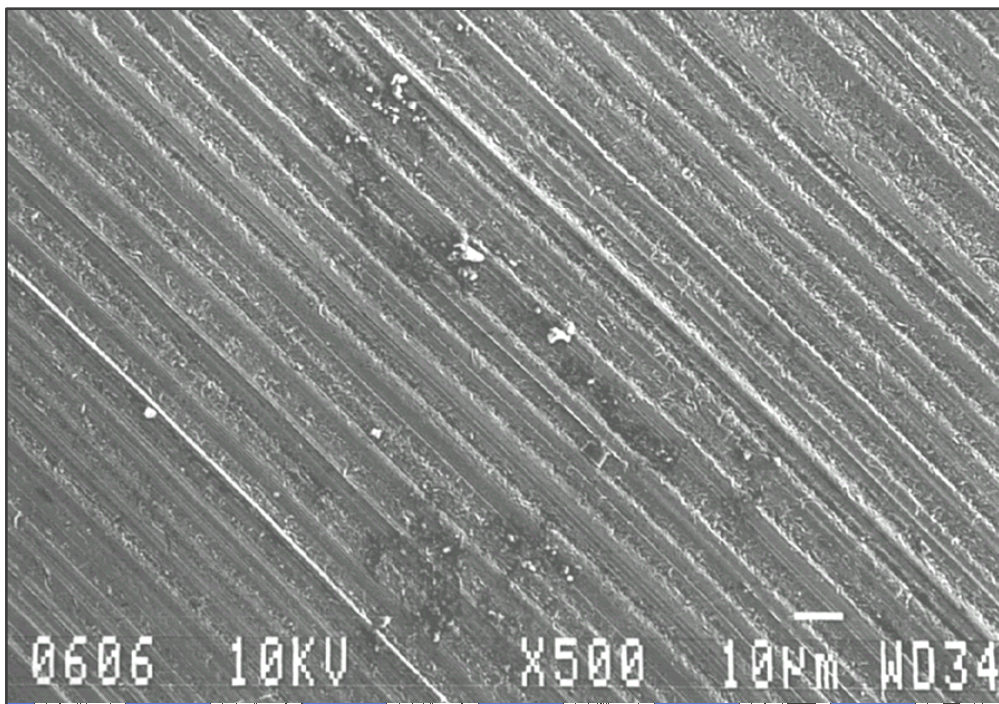


Figure 18 Ball cratering wear mechanism map for tool steel sample with SiC abrasive, ref [23]



a)



b)

Figure 19 Worn surfaces of tool steel samples tested with SiC abrasive a) in rolling wear regime, b) in grooving wear regime, ref [23].

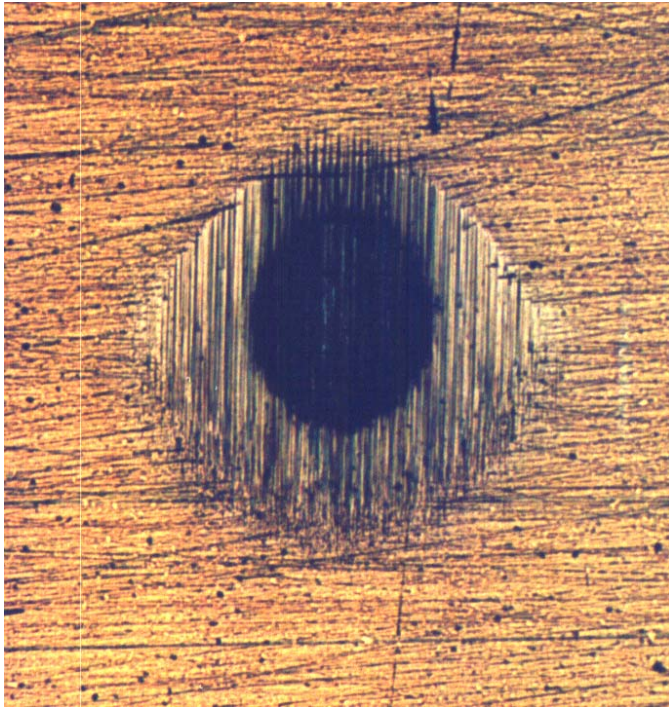


Figure 20 Overall appearance of TiN crater tested in grooving wear regime.

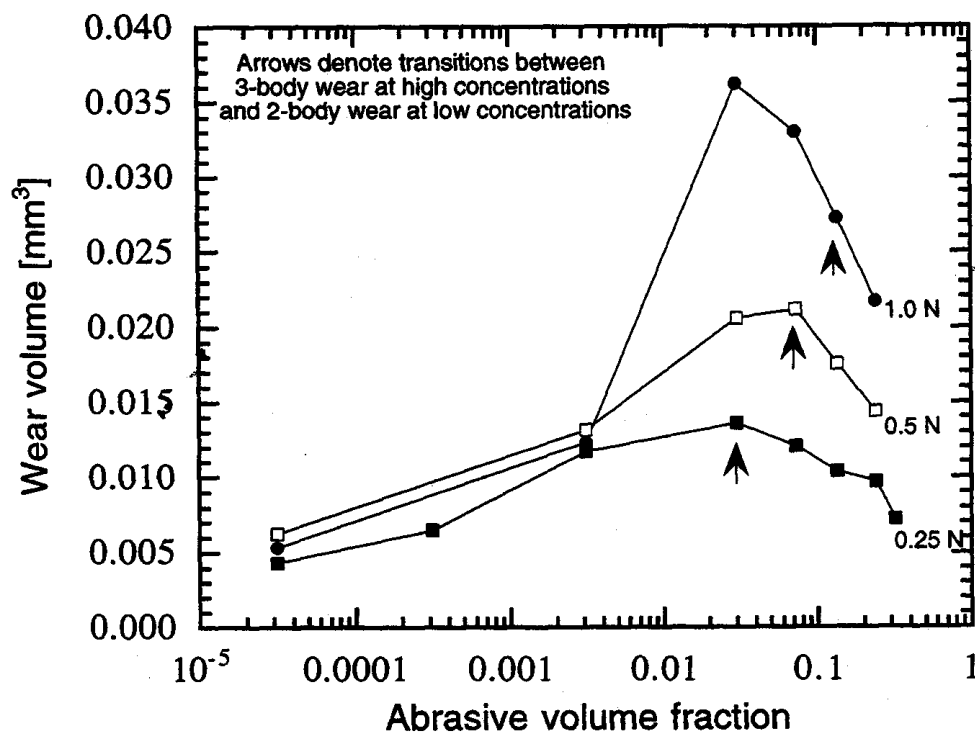


Figure 21 Variation in magnitude of wear of tool steel sample with SiC abrasive slurry concentration, ref [23].

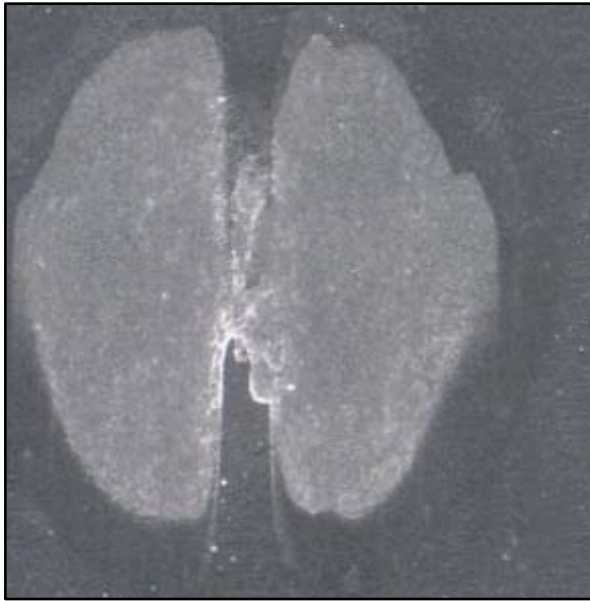


Figure 22 Ridge formed at centre of crater on TiN coated tool steel sample under high load dry sliding conditions.

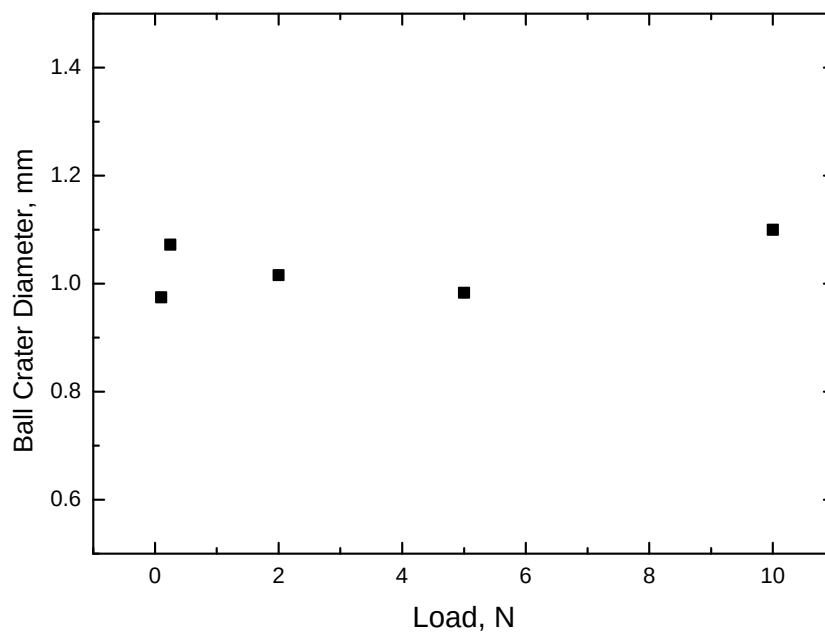


Figure 23 Lack of variation of wear with changing load for WC/Co sample tested with SiC abrasive. Lack of wear is correlated with increasing size of ridge.

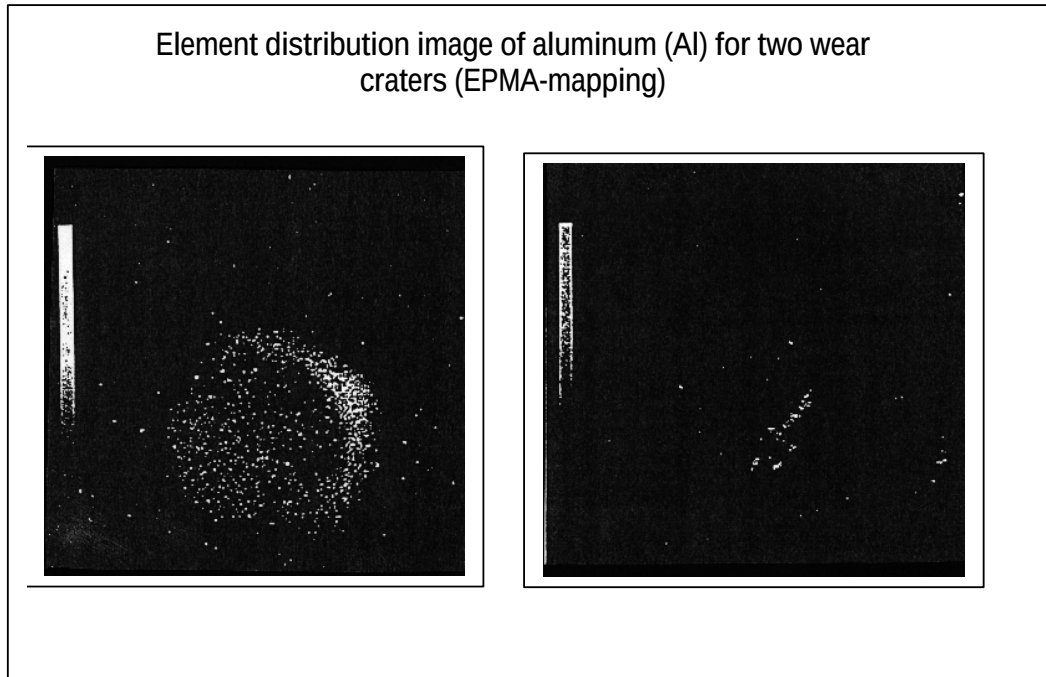


Figure 24 Embedding of alumina abrasive in the coating by high loading on left, no embedding on right with appropriate loading [13].

9.3 Suspension Fluid

Clearly, if the composition of the slurry suspension fluid promotes corrosion or chemical reaction with the sample, a synergistic effect of variation of the wear rate is likely to occur. Although there is some evidence that the physical properties of the fluid such as viscosity may have an impact on results [13] for high viscosity fluids such as glycerine, for many practical suspension fluids (eg water), the speed of relative motion between the ball and the sample is sufficiently low that hydrodynamic lubrication effects are not important.

There may be lubrication effects due to changes in the way the suspension fluid lubricates the contacts between the ball, abrasive particle and sample, but there is currently no clear evidence for this.

There is conflicting evidence over the effect of suspension dosage. Some workers suggest that as long as there is always sufficient suspension at the wear interface there will be no effect on results, whilst others report a high influence and state the need to dose the slurry precisely [13].

9.4 Sliding Speed

The wear volume (at constant sliding distance) is largely independent of sliding speed, but increases somewhat for very low speeds (Figure 25) [15].

For the free ball machine, the tendency of the ball to slip on the shaft increases as the speed increases. This can lead to an apparent decrease in wear as the ball increases (Figure 26) [13].

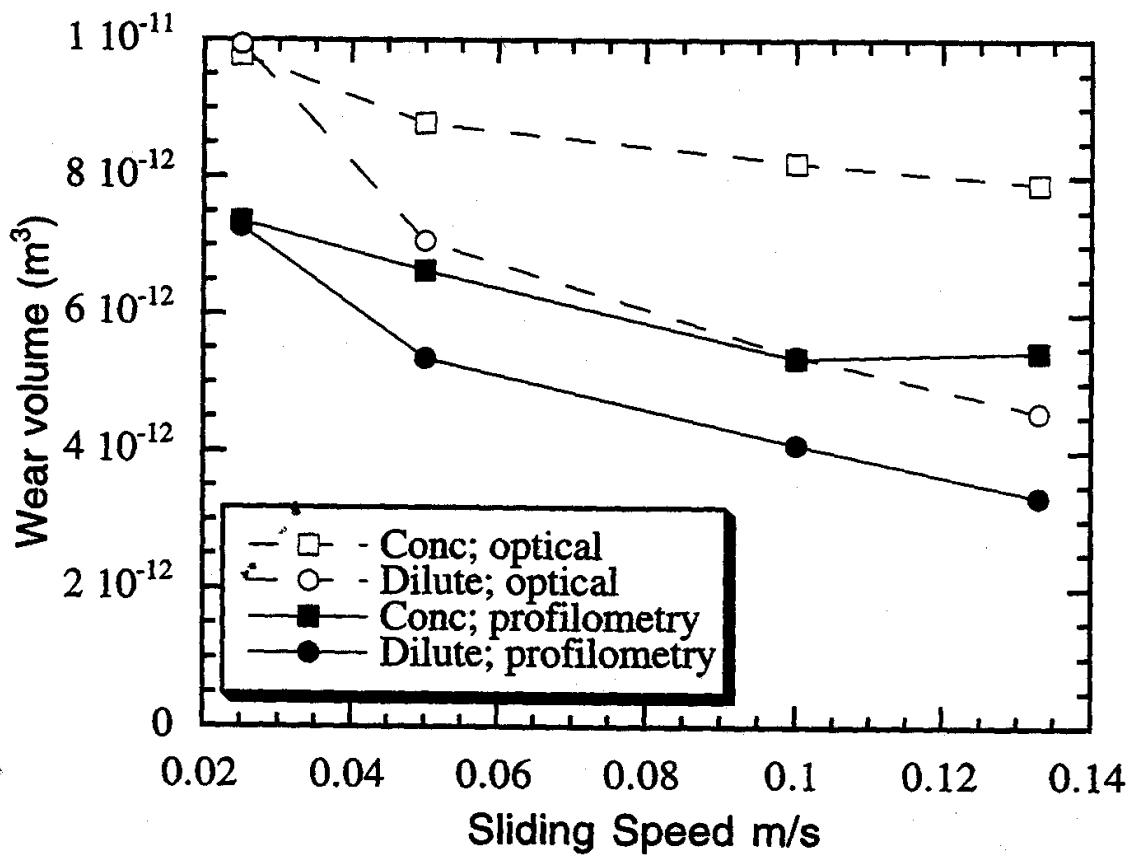


Figure 25 Variation in wear volume for tool steel sample with sliding speed, ref [18].

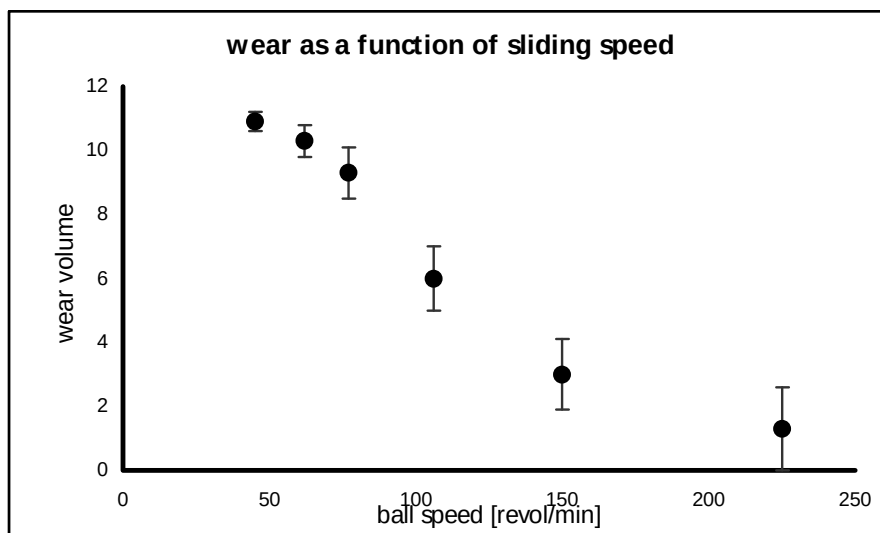


Figure 26 Effect of speed on apparent wear for free ball-single shaft machine with DLC coating and 1 µm diamond suspension [13].

9.5 Ball Material

There are two effects of changing the ball material; both due to the changes in the relative hardness. Thus a softer ball will encourage embedding of abrasive particles into the ball, reducing separation between the surfaces and enhancing grooving wear.

If very soft balls are used, the normal assumption in the calculation of wear volumes that the wear scar is conformal to the unstressed shape of the ball may not be true. The ball deforms under the applied load so that the contact area becomes much larger than would be otherwise expected. This makes analysis of test results much more complex.

Unless there are special reasons for using other materials, most users use hardened steel balls.

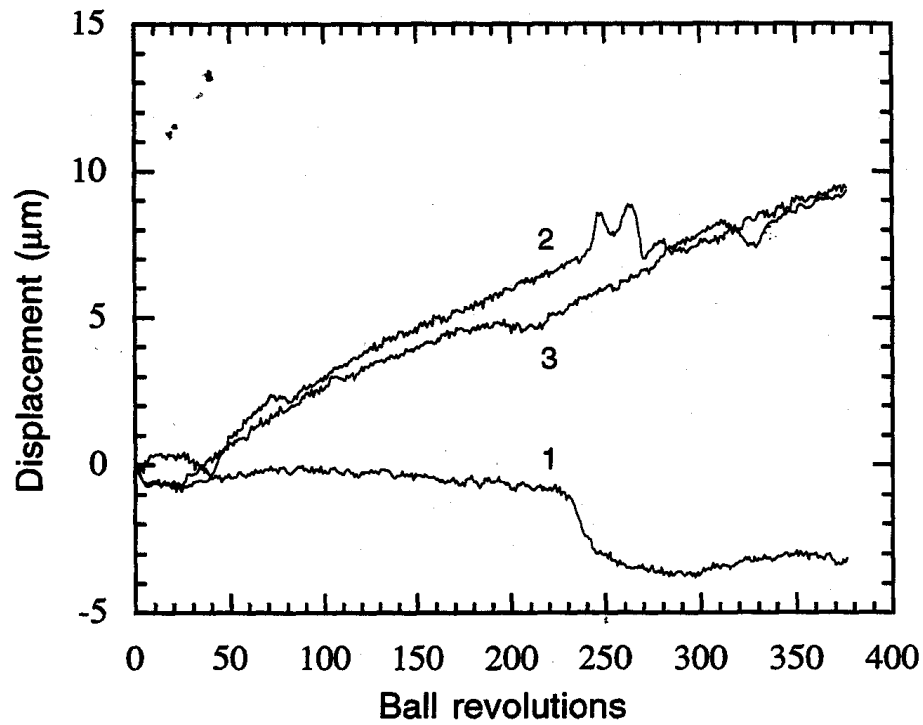
9.6 Surface Ball Condition

The condition of the test ball has been shown to have a major effect on the wear that occurs. As balls are used in tests, they become roughened through abrasion and potentially corrosive damage.

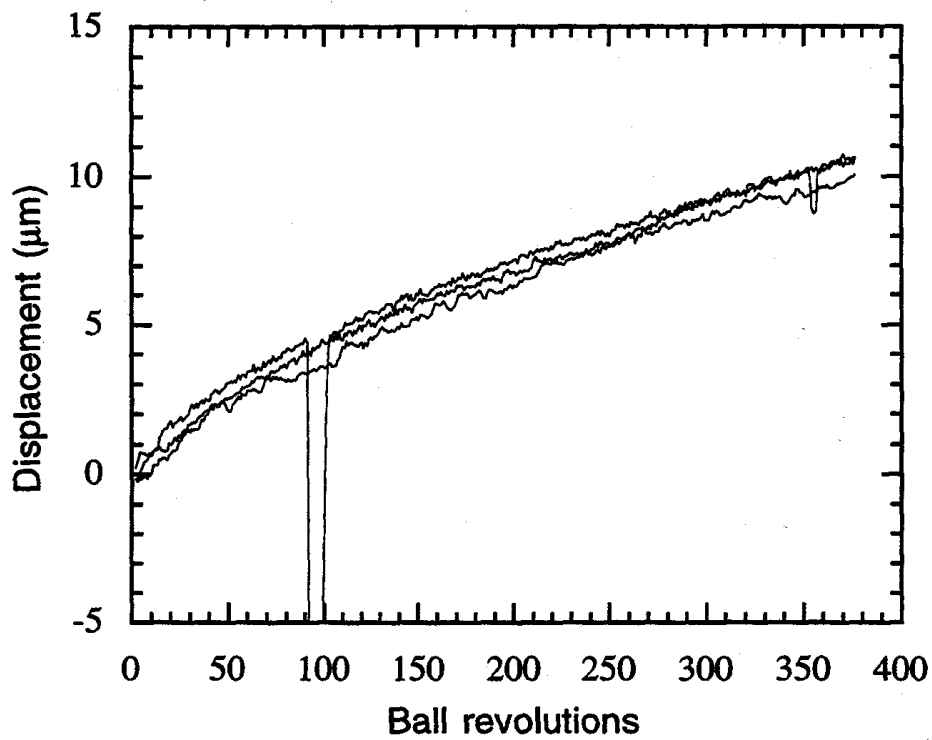
In experiments where the wear displacement was measured as a way of monitoring wear on-line, the wear obtained with used balls was compared with the wear obtained with new balls [24]. It was found that the wear displacement for the worn balls was more reproducible than with the new ball. This was confirmed to be due to a lack of entrapment of abrasive particles with the new balls. As the test proceeds, and the ball gradually roughens, abrasive becomes entrapped in an irregular random fashion. With the rougher used balls entrapment occurs almost immediately the test starts.

Although surface treatments have been developed for the balls [24] to eliminate the variability introduced by this effect, perhaps a better procedure is to condition new balls by carrying out a “running in” procedure on the new balls on a sacrificial part of the sample.

As well as reducing variability in results, the use of treated or used balls increases the wear that is observed (Figure 28). However, as wear to the test ball continues, a flat band can be worn on the ball giving erroneous results. It is therefore important to inspect the ball at regular intervals and replace it if it becomes too worn.



a)



b)

Figure 27 Wear displacement traces for three repeat tests on silver steel with SiC abrasive, a) new test balls, b) used test balls, ref [24].

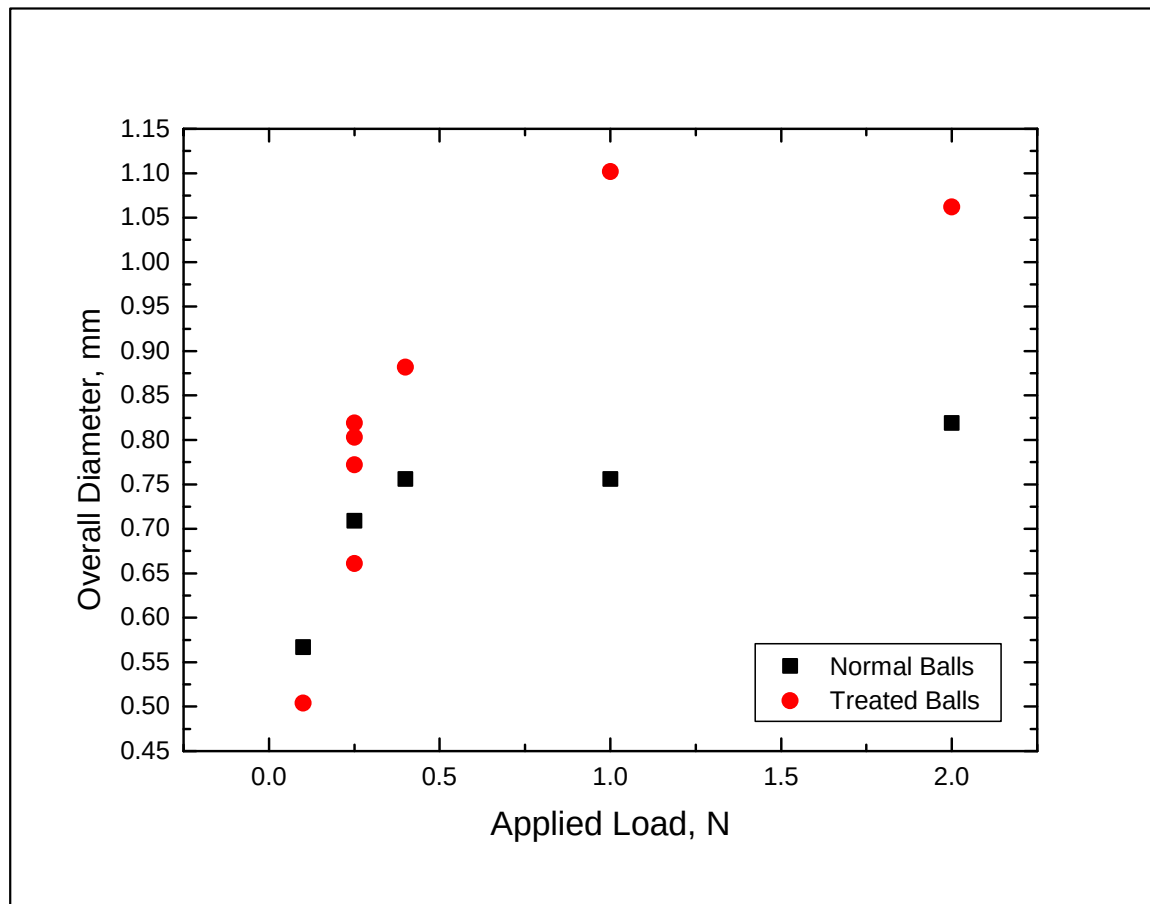


Figure 28 Increase in wear for treated balls by comparison with new balls. Tests on TiN coated tool steel with SiC abrasive.

10 Factors Affecting Measurements

Factors that affect measurement are:

- the difficulty in resolving the edges of craters
- the method of scar size measurements
- the fact that non-perfect crater slopes are often seen in tests
- the theory for analysing results may not be appropriate for all materials and test conditions.

10.1 III Defined Crater Edges

Often there can be difficulties in defining the edges of craters. Figure 29 shows the edge of a crater made in a TiN coating on tool steel. Grooving wear took place in this test, but some of the grooves were so deep that it made measurement of the inner crater diameter impossible in this case (also see Figure 17). This Figure (29) also shows that there is a region of scuffing that has occurred around the edge of the crater. This scuffing can cause difficulties in determining the real edge of the crater. Thus in Figure 30, optical microscopy can be seen to overestimate the size of the craters by comparison to direct profilometric measurement of wear volume [18] (look at the gap between the two craters when comparing the two measurement methods). A similar result can be seen in Figure 31 [13] where the wear rate calculated from optical measurements is larger than that calculated from profilometry measurements.

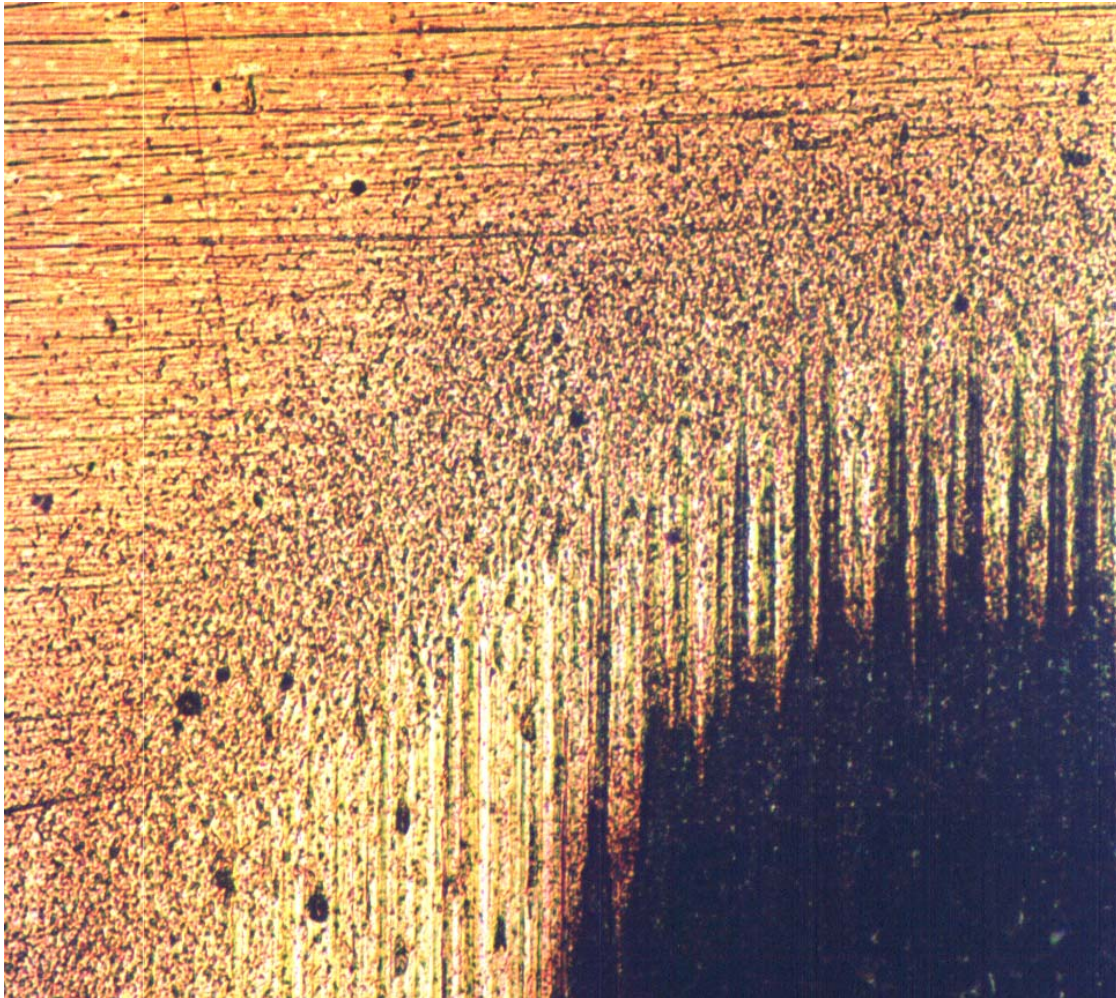
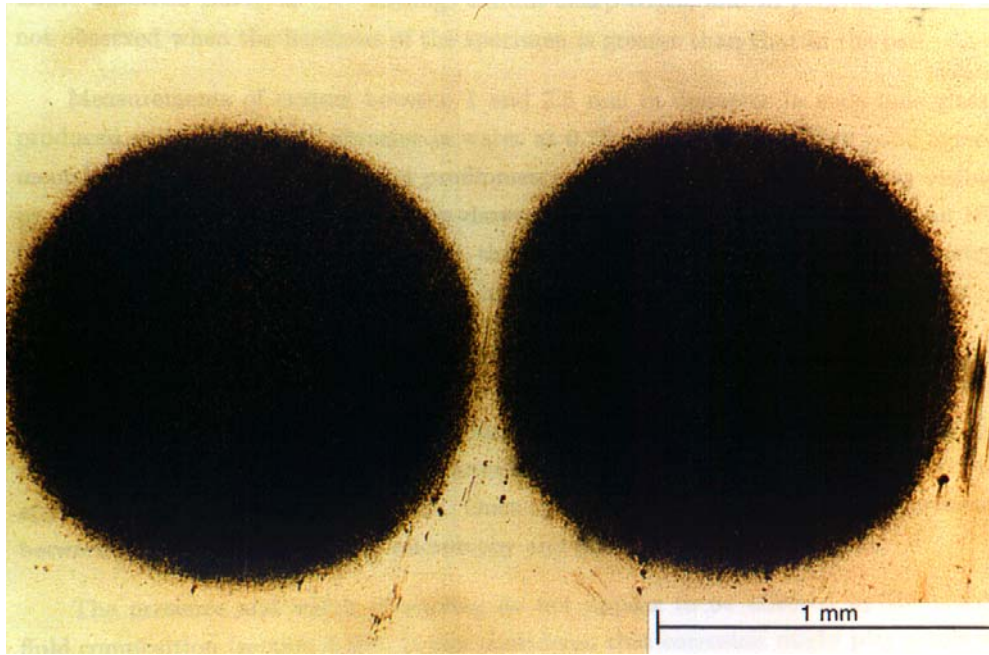
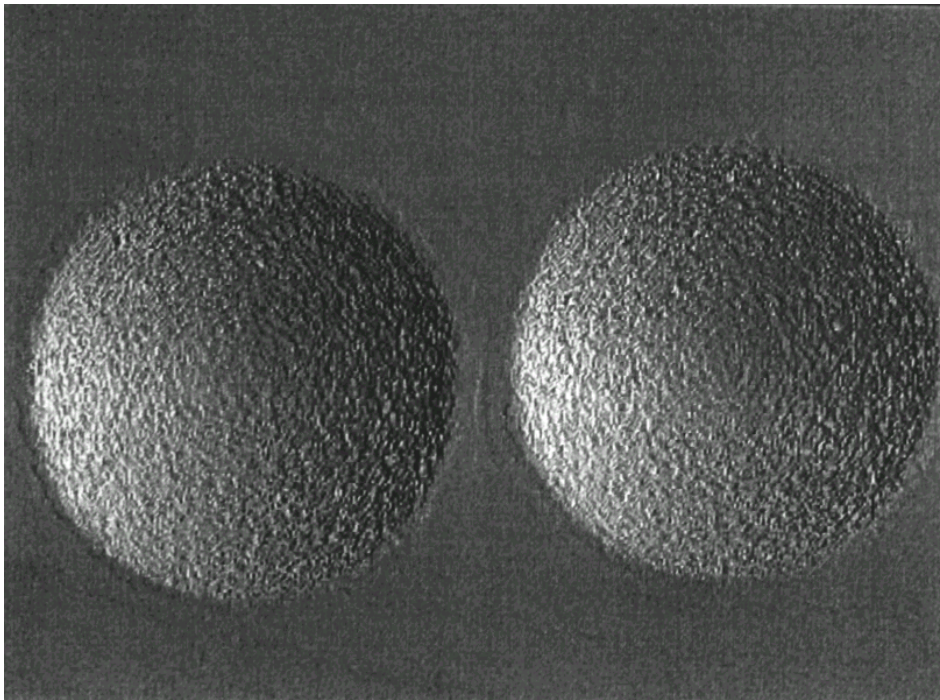


Figure 29 Poorly defined edge of crater.



a)



b)

Figure 30 Twin (same test conditions) craters on tool steel sample, a) optical micrograph, b) height map from profilometric measurement, ref [18].

10.2 Different Measurement Methods

The different measurements that have been used for crater size measurement are:

- optical microscopy through image analysis or travelling microscopy
- profilometry through interference microscopy, laser optical techniques, or mechanical probe devices.

The use of profilometry sometimes overcomes the difficulties mentioned in the last section, but clearly the boundary between the portion of the crater in the coating, and the portion in the substrate cannot be distinguished by this method, as profilometry can only be used for testing where perforation does not occur.

10.3 Reproducibility

The ball cratering instrument has been tested using standard wear protective coatings e.g. DLC, W-C:H and TiN. The results for W-C:H and TiN are shown in Figure 32 [13]. The sliding distance (number of revolutions) has been adjusted in a way that the wear process is limited to a depth of 1 μm , where the wear properties of the coating could be measured without influence of the substrate or interlayer. The measurements show high reproducibility of the wear depth measurement with a standard deviation of less than 5%.

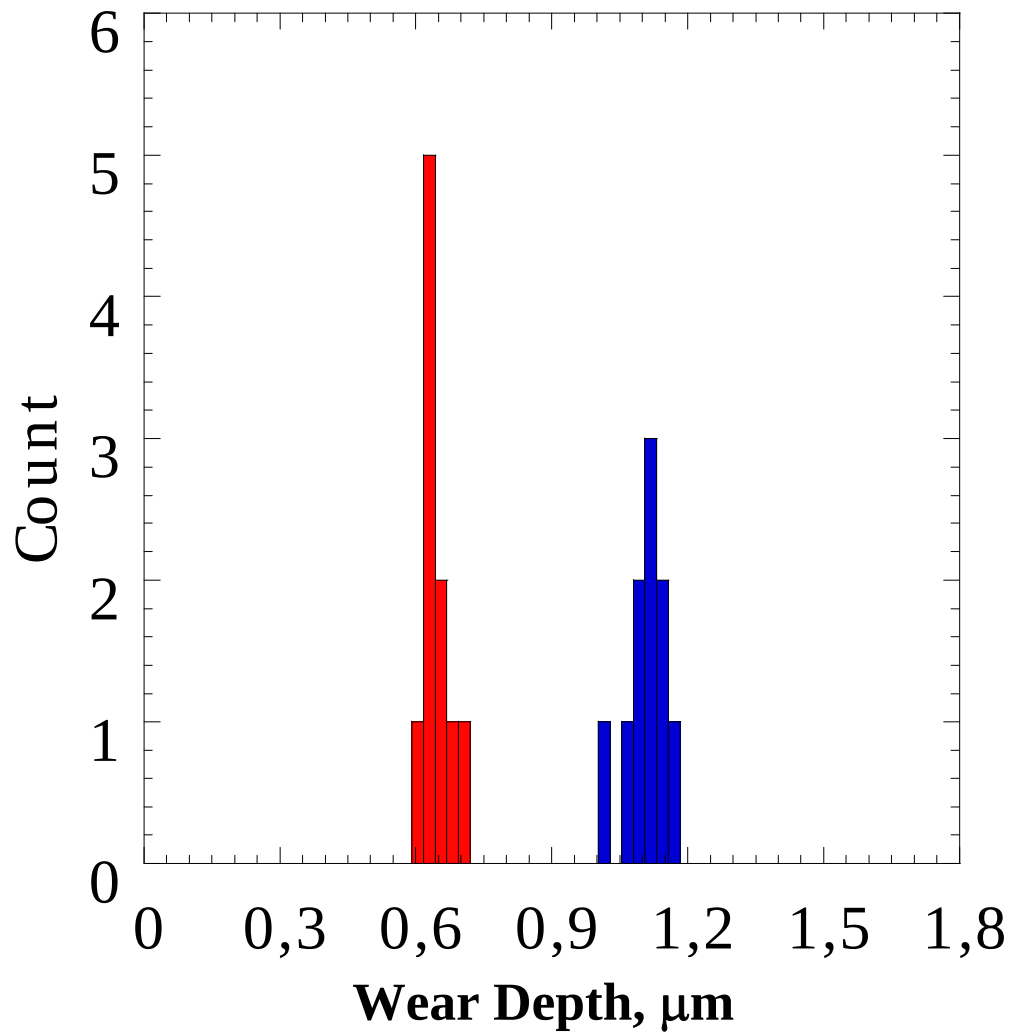


Figure 31 Repeatability of non-perforating ball crater tests on free-ball-single shaft system [13]

10.4 Comparison between perforating tests and non-perforating tests

It is useful to compare the two modes of use of the ball cratering test. Clearly for thick coatings (more than 10 μm in thickness), the non-perforating test will be most useful as perforation for these coatings will take a long time. In this situation, good measurement accuracy can be achieved without the need to make the assumptions that lie behind the analysis for the perforating test that may not be valid in all cases. For thin coatings (less than 1 μm) the coatings are perforated very quickly and the only possible route is the use of the perforating test. However, as the coating becomes thinner, it becomes increasingly difficult to measure and differentiate the relative sizes of the inner and outer craters, so accuracy is a concern.

Between these two limits either type of test and the relative merits of both types of test will vary with the materials that are being tested.

A systematic comparison between perforating and non-perforating tests has not yet been made; this is one of the expected outcomes of the CRATER EU project.

11 Sources of Guidance

The first part of these guidelines has given a simple introduction to the different mechanisms of wear, followed by consideration of the ball cratering test for the abrasive testing of coatings. There are a number of books and other documents which are available if more information is required: Some sources of guidance (which are by no means exhaustive) are listed here:

- A useful introduction in the consideration of the framework for friction and wear measurement is given in *Tribology: a systems approach to the science and technology of friction lubrication and wear* by H Czichos [25]
- General information on tribology is available in several handbooks such as the *Wear Control Handbook* by Peterson and Winer [26], *The ASM Metals Handbook: Vol. 8, Friction, Lubrication and Wear Technology* [27], and the *Handbook of Tribology: Materials, Coatings and Surface Treatments* by Bharat Bhushan and B K Gupta [28]
- Compilations of data of wear rates of different materials combinations under specified conditions are available in the *Tribology Handbook* by M J Neale [29] and the *Elsevier Materials Selector* by N A Waterman and M J Ashby [30].
- Specific information of testing is given in many papers in the literature. Example are Almond and Gee [31], Alliston-Greiner [32], Eyre [33], in ASTM STP's on friction and wear testing of metals [34], plastics [35], coatings [36], elastomers [37], ceramics [38], and advanced materials [39], and an ASTM STP on the fretting fatigue test methods [40].
- Other more general information on wear testing is given in books such as *the Fundamentals of Friction and Wear of Materials* edited by Rigney [41], *New directions in Lubrication, Materials, Wear and Surface Interactions* by Loomis [42], *Tribology: Friction and Wear of Engineering Materials* by Ian Hutchings [43], *Engineering Tribology* by John Williams [44], and *Friction Science and Technology* by Blau [45].
- Many papers on friction and wear testing procedures are given in the proceedings of conferences such as the biennial *International Conference on the Wear of Materials* [46], and the peer reviewed journals *Wear* [47] and *Tribology International* [48].
- Practical and comprehensive guidance is also given in the *Guide to Wear Problems and Testing for Industry* [1], by Neale and Gee.

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13 Annex A: Procedure for Ball Cratering Test

1 BACKGROUND

This procedure describes the method for performing tests with the ball cratering test system that has been developed for carrying out wear tests on thin hard coatings [1,2].

The procedure builds on work carried out both as part of the EC funded FASTE project on the Development and Validation of Test Methods for Thin Hard Coatings. The procedure will be validated through an EU funded project, through a DTI funded project, and also through the VAMAS Technical Work Area 1: Wear Test Methods.

2 TEST SYSTEM

The test system can either use a ball or a profiled wheel which is rotated and pressed against the coated sample. When a wheel is used the sample must also be rotated. Two different variants of the ball system are shown in Figure 1, where either the sample mounted on a dead-weight loaded lever is pressed against a directly driven ball, or the ball's own weight presses it against the sample.

In all cases, a slurry of SiC abrasive in water is drip fed onto the contact between the ball and the sample and wear is measured by measuring the size of the wear scar on the sample using a microscope.

3 MATERIALS

The coated samples should have a flat area large enough to perform the necessary series of experiments. The coating thickness can be evaluated as part of the test procedure, but must be larger than 1 μm . The balls are 25 mm diameter hardened steel (AISI 52100).

Balls can be used in a polished condition, but it has been found [3] that the test behaviour is erratic and poor results are obtained if balls of this type are used without conditioning.

The conditioning treatment consists of running the test ball for 50 revolutions on a non-critical part of the sample under normal test conditions before starting the testing.

The abrasive is normally F1200 SiC abrasive, but F1200 silica or other fine abrasive can be used. The average size of the abrasive should not exceed 4 μm . An abrasive slurry should be made up from the abrasive powder and distilled water in the required proportions.

As the mode of wear that is observed depends critically on the abrasive concentration that is used, two concentrations are recommended. These are:

1) Dilute (promotes grooving wear)

Concentration of 2 % by volume

For SiC this is achieved by mixing 6.4 g to 98 ml distilled water.

2) Concentrated (promotes rolling wear)

Concentration of 20% by volume

For SiC this is achieved by mixing 6.4 g to 8 ml distilled water

As an alternative to mixing slurries, ready mixed abrasive slurries can be used. If this is done all details of the supplier and makeup of the slurry should be reported.

4 TEST TYPES

Two different types of tests can be performed.

Type A: No perforation of coating.

In this type of test the duration of the test is controlled so that perforation of the coating does not occur. Some trials may be necessary before the required conditions are obtained. The wear is calculated from the measured size of the wear crater.

Type B: Perforation of coating.

In this type of test the coating is perforated. A series of craters with different durations is produced and the sizes of the craters are measured in each case. The wear rates for both the substrate and the coating can be calculated from an analysis of the measured crater sizes.

5 TEST CONDITIONS

1. Number of test repeats

Two repeat sets of tests should be carried out on the sample.

2. Load

If the load applied to the sample is too high the phenomenon of ridging occurs. To prevent this, it is suggested that the load applied should not be greater than 0.4 N. The recommended load is 0.25 N.

3. Speed

The speed of the ball should be controlled at a constant value throughout a set of tests. A recommended speed is 80 rpm.

When a wheel test system is used the sample rotation speed should be reported.

4A. *Number of revolutions: Test type A with no perforation*

The number of revolutions required to give a crater which is easy to measure, but with no perforation needs to be determined by trials.

4B. *Number of revolutions: Test type B with perforation*

The number of revolutions that is required will depend on the materials that are to be tested and the test conditions employed. However, a set of measurements for 100, 200, 300, 600 and 1000 revolutions has been found to be appropriate for tests using a concentrated SiC abrasive slurry under a load of 0.25 N and at a speed of 80 rpm.

6 TEST PROCEDURES

Type A: No perforation of coating.

1) The sample should be cleaned by the following procedure:

Ultrasonically clean in acetone for 15 minutes
Rinse in clean acetone
Dry in oven at 110 °C for 10 minutes.

2) The sample should be clamped firmly into position on the test system.

3) The motor speed should be adjusted to give the correct speed.

4) The test system should be adjusted to give the correct normal loading between the ball and sample at a test point on the sample.

Note: In free ball machines, the friction due to the ball rotation results in an error in the normal force acting on the sample. In these test systems a load cell should be employed to measure the true normal force.

- 5) The slurry drip feed should be started, with the slurry continuously stirred by a magnetic stirrer. The feed rate should be sufficient that the contact between the ball and sample is always well wetted by the slurry. The slurry should not be recirculated.
- 6) After the required test duration, the test should be stopped (motor and slurry feed stopped).
- 7) The sample should be removed and cleaned by the same procedure as in 3).
- 8) The size of the wear scar should be measured both parallel and perpendicular to the direction ball rotation. Both the full crater width, and the width of the substrate crater should be measured (Figure 2). The average of these measurements should be used as the sizes of wear scar b and a in the analysis described below.
- 9) The wear rate of the coating should be calculated using the method described below.

Type B: Perforation of coating.

Steps 1-6 are the same as in the type A test.

- 7) When the required number of revolutions has taken place for the first crater, the test should be stopped (motor and slurry feed stopped), the sample moved so that a new position on the sample is worn and the next test in the sequence performed.
- 8) When the sequence of tests has been completed, the sample should be removed and cleaned by the same procedure as in 3).
- 9) The size of the wear scars should be measured both parallel and perpendicular to the direction ball rotation. Both the full crater width and the width of the substrate crater should be measured (Figure 2). The average of these measurements should be used as the sizes of wear scar b and a in the analysis described below.
- 10) The wear rate of the coating and substrate should be calculated using the method described below.

- 11) The thickness of the coating at the test position can also be calculated as described below

7 ANALYSIS OF RESULTS

Type A: No perforation of coating.

The volume of wear is given by:

$$V = \pi \frac{b^2}{64R} \quad \text{for } h \ll R$$

where R is the radius of the ball and b is the crater diameter. The Archard wear equation relates the volume of wear to the normal load N and the sliding distance S as:

$$V = K_C SN$$

where K_C is the wear coefficient for the coating. Thus

$$K_C = \pi \frac{b^4}{64RSN}$$

Type B: Perforation of coating.

The basic equation which is assumed to govern wear is (1)

$$SN = \left(\frac{V_c}{K_c} + \frac{V_s}{K_s} \right) \quad (1)$$

where S is the distance slid by the sphere, N is the normal load, V_c is the volume of coating removed, V_s is the volume of substrate removed, K_s is the wear coefficient for the substrate, and K_c is the wear coefficient for the coating.

Equation (1) can be expressed as

$$SN = \left(\frac{V_c K_s + (V - V_c) K_c}{K_c K_s} \right) \quad (2)$$

where V is the total wear volume of the crater.

The total volume of the crater is given by

$$V = \pi \frac{b^4}{64R} \quad \text{for } h \ll R \quad (3)$$

where b is the external diameter of the crater, R is the radius of the ball and h is the depth of the scar, and the volume of the worn coating is given by

$$V_c = \pi t \left(\frac{b^2}{4} - Rt \right) \quad \text{for } h \ll R \quad (4)$$

where t is the thickness of the coating (calculated by the equation given in the Annex from the measurements of the scars).

Rearrangement gives

$$\frac{SN}{b^4} = \left(\frac{K_s - K_c}{K_s K_c} \right) \left(\frac{\pi t}{4b^2} - \frac{\pi R t^2}{b^4} \right) + \left(\frac{1}{K_s} \right) \left(\frac{\pi}{64R} \right) \quad (5)$$

Thus a plot of SN/b^4 against $(\pi t/b^2 - \pi R t^2/b^4)$ should be linear with the intercept as $\pi/64RK_s$ and the slope of $(K_s - K_c)/K_s K_c$.

The values of b , a and t should be calculated for each individual scar, and a regression analysis performed of SN/b^4 against $(\pi t/4b^2 - \pi R t^2/b^4)$.

If the intercept is C and the slope M , then

$$K_s = \frac{\pi}{64RC} \quad (6)$$

and

$$K_c = \frac{K_s}{K_s M + 1} \quad (7)$$

8 REPORTING

The details of the materials should be clearly described.

The type of test that has been performed shall be given.

All test results should be reported giving full details of all values of scar diameter measurements, and the duration of test in revolutions when the measurements were obtained.

9 REFERENCES

- 1) A Kassman, S Jacobson, L Erickson, P Hedenqvist and M Olsson, *A new test method for the intrinsic abrasion resistance of thin coatings*, Surf. Coat. Technol., **50** (1991) 75-84.
- 2) K L Rutherford and I M Hutchings, *A micro-abrasive test, with particular application to coated systems*, Surf. Coat. Technol., **79** (1996) 231-239.
- 3) D N Allsop, R I Trezona and I M Hutchings, *The effect of ball surface condition in the micro-abrasive wear test*, Tribology Letters, **5** (1998) 259-264.

Figures

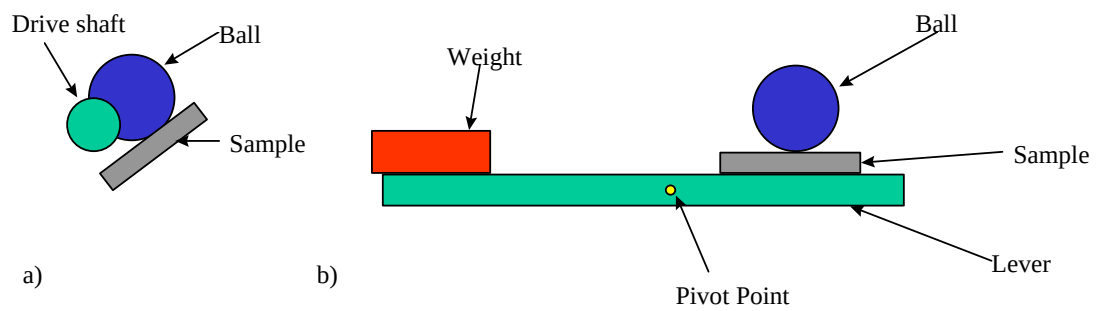


Figure 1, Two different types of ball cratering system, a) free ball, b) fixed ball.

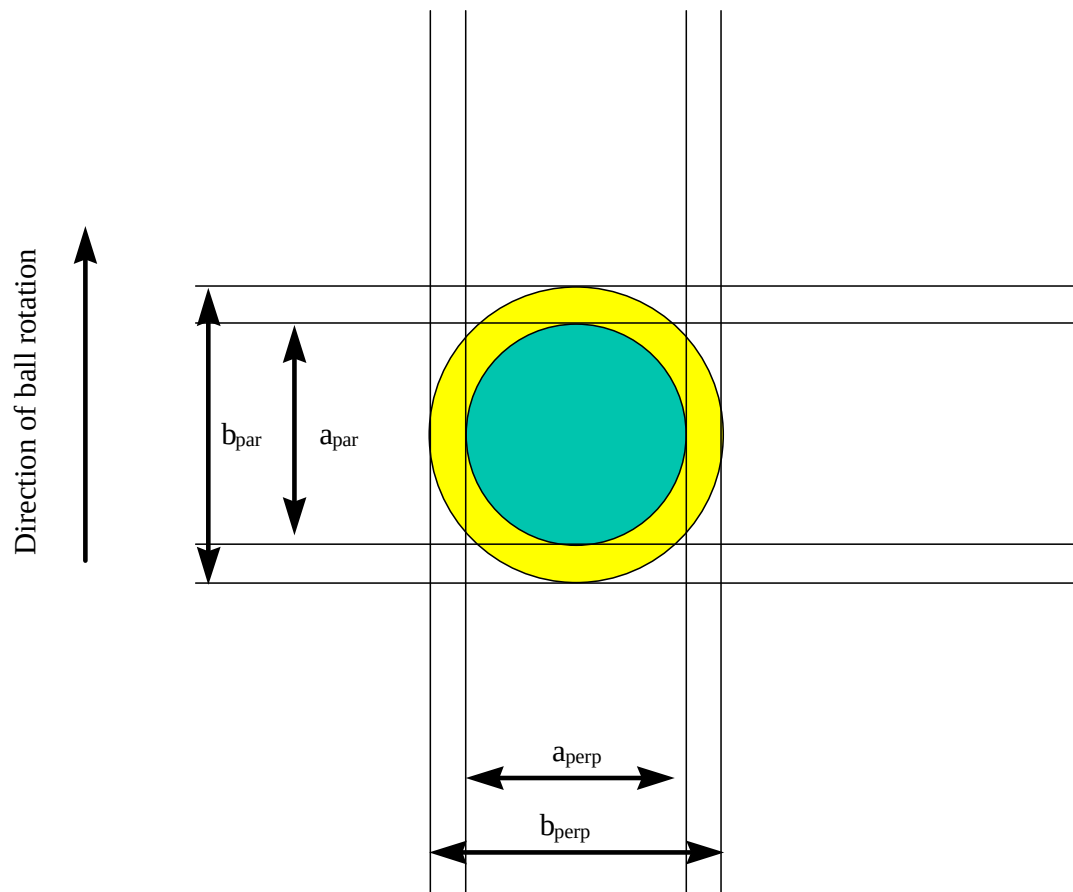


Figure 2, Measurements on wear scar

14 Annex B: Measurement of Coating Thickness

The coating thickness t used in the analysis for the wear rates above should be calculated from the measurements of scar diameters made in the tests.

If b is the overall crater diameter and a is the diameter of the crater in the substrate, then t is given by:

$$t = \left(R^2 - \left(\frac{a}{2} \right)^2 \right)^{\frac{1}{2}} - \left(R^2 - \left(\frac{b}{2} \right)^2 \right)^{\frac{1}{2}}$$

The average of the values calculated with this equation should be used in the calculation of wear rates.