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Results from an interlaboratory exercise to validate the micro-scale abrasion test

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Abstract

The mico-scale abrasion test has become very popular in recent years for the measurement of the abrasive wear of coatings and other materials. A EU-funded project has just been completed that had the aim of developing the test method, and revising the existing CEN ENV for the test into a full standard.

Key aspects of the project were concerned with the accuracy of measurements, the repeatability and reproducibility of the test method through an interlaboratory exercise, and the industrial applicability of the measurement method to different types of coatings.

The main measurement methods that can be applied are optical measurements and profilometry. Here, it was found that profilometry measurements gave important information on the shape of the crater produced during the test, but optical measurements were found to be adequate in most cases. The reproducibility of the optical measurements was found to be about 2% when the results of different laboratories were compared.

Fourteen organisations participated in the interlaboratory exercise. This found reasonable reproducibility and repeatability for the measurement method. The paper concludes by describing the procedure that was recommended at the end of the project, and is likely to be adopted in CEN, ASTM and ISO standards.

Keywords: Mico-scale abrasion test; Interlaboratory exercise; Measurement

1. Background

The use of coatings and surface engineered components is now growing strongly. This is being driven by the need for high value added products with enhanced durability, efficiency and lower environmental impact. These coatings and surface engineered components are used in all sectors of engineering including, for example, aerospace engines and components, prosthetic implants, paint films on cars, internal combustion engines and textile machinery.

The micro-scale abrasion test is a promising technique that has the potential to assess the wear resistance of coated and surface engineered components [1,2]. The test originated from the technique of using rotating balls to produce taper sections for surface analysis [3]. The background literature

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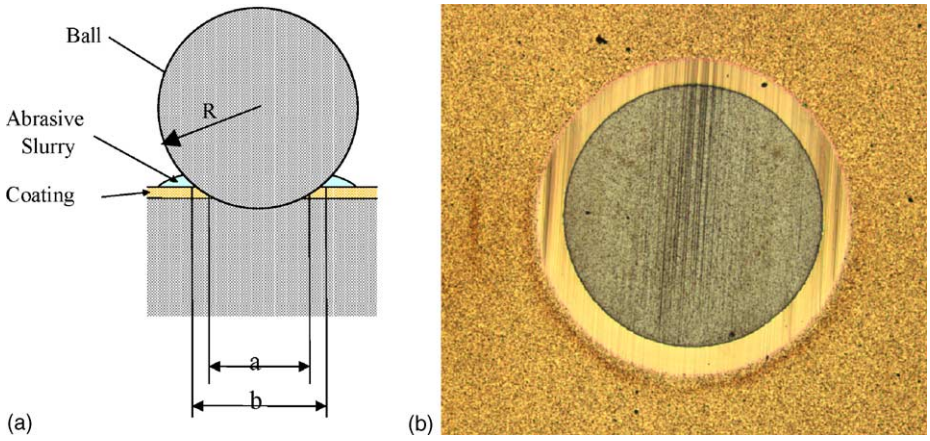

Fig. 1. Micro-scale abrasion testing: (a) schematic and (b) crater produced on TiN coating.

to the test method is reviewed in an earlier publication [2], and so is not discussed further here.

The test method can be used to evaluate all types of coating including polymeric films, thin hard ceramic coatings, metallic coatings and thick thermally sprayed coatings.

In the technique, a ball often of bearing steel, is pressed against the test sample in the presence of an abrasive slurry (Fig. 1a). This produces a circular depression in the sample that can be measured (Fig. 1b) to determine the wear rate of the coating, and under some conditions the substrate material.

There are two variants of the test. In the non-perforating test, wear is confined to the coating alone and a wear rate is calculated by simple geometrical considerations. In the perforating test, a sequence of craters is made of different durations that penetrate the coating producing two boundaries marking the overall crater diameter and the portion of the crater in the substrate. Analysis of the size of these features yields wear rates for both the substrate and coating.

The test technique has now been investigated in a 3-year project, funded in part by the EU that had 10 partners from across Europe. The parameters that affect the test results

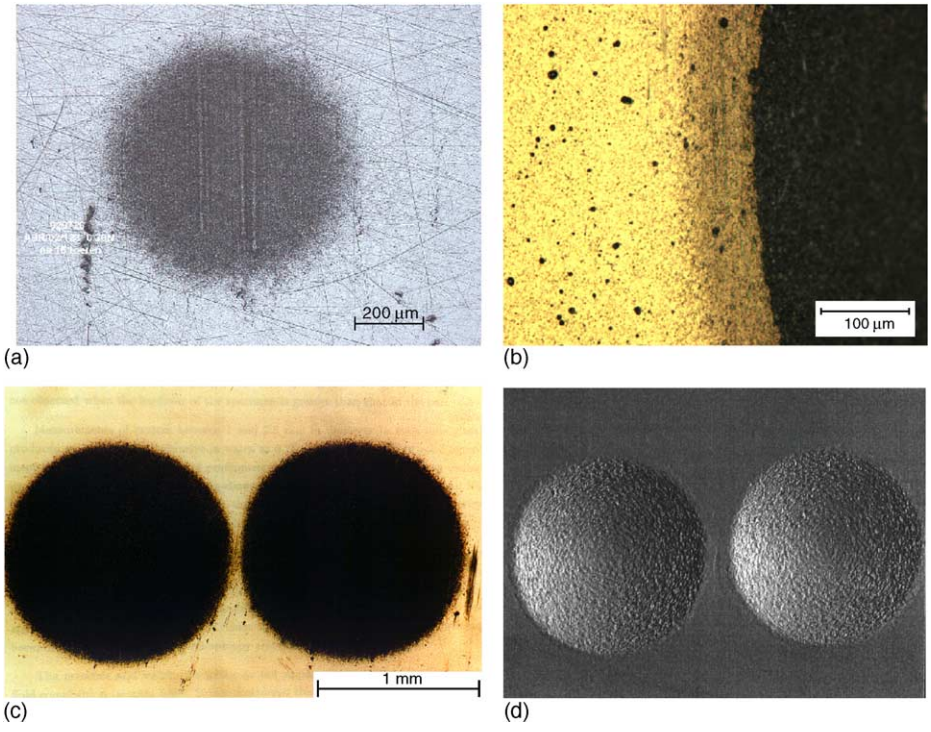

Fig. 2. Optical micrographs showing (a) scuffing at edge of small crater on DLC coating, (b) difference in contrast for inner and outer crater boundaries, (c) optical micrograph of pair of craters made on tool steel sample, (d) profilometric images showing real shape of same craters as in (c). Note the apparent separation between the two craters comparing (c and d).

have been thoroughly examined, the accuracy of measurement methods assessed, and the reproducibility of the test has now been evaluated by performing an interlaboratory exercise.

2. Measurement issues

The main purpose of this paper is to present the modified test procedure developed in the EU project, and the results of the interlaboratory exercise. For this reason, only a few of the main results from the other parts of the EU project will be described here. For further information, the reader is referred to the final report of the project [4].

One of the main conclusions was that the measurement of the overall crater diameter could be difficult with the normal SiC abrasive used in micro-scale abrasion tests. This is due to phenomena such as scuffing or rounding at the edge of the outer crater. In Fig. 2a, the indistinct edge of a small crater made on a DLC coating can be seen; in Fig. 2b the contrast at the outer edge of a crater on a TiN coating is less than for the inner crater, and comparison of Fig. 2c and d shows that in this case, the optical measurements over estimate the size of the craters in tool steel. Other profilometric measurements confirm rounding of the outer crater diameter, but also confirm that the craters conform well to the shape of the ball [5]. For this reason, a new measurement and analysis procedure has been introduced in the EU study [6], where the thickness of the coating is measured independently using the cap grinding technique [7] using a fine diamond abrasive to ensure that scuffing and rounding do not occur.

Although profilometric measurement provides considerable useful information about the size and shape of craters, it was decided that for routine measurements optical techniques of the inner crater diameter would normally be used when combined with the independent measurement of the coating thickness mentioned above. To examine the reproducibility of optical measurements samples of TiN coated tool steel was prepared by Cambridge University and sent out to the different project partners. The results shown in Fig. 3, expressed as the ratio of the individual partners measurements to the measurements at Cambridge University, show that the uncertainty in measurement is about 2%.

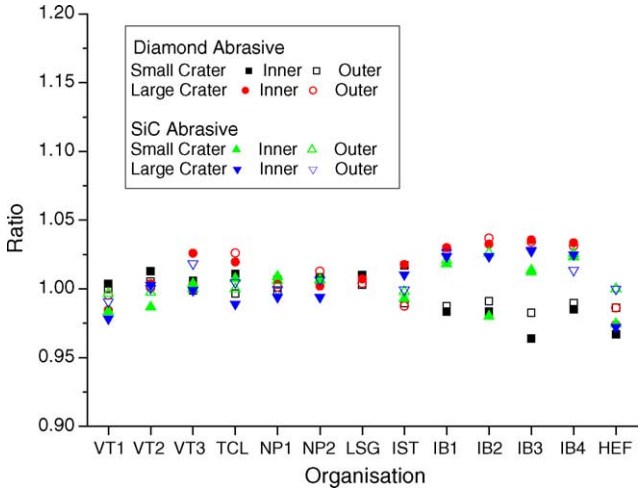

Fig. 3. Reproducibility of measurements of craters prepared by fine diamond and SiC abrasive on TiN coated tool steel sample. Seven organisations participated, in some cases using different microscopes denoted by different numbers in figure.

3. Interlaboratory exercise

3.1. Materials

The test samples were ASP23 powder metallurgy steel 6 mm thick × 32 mm in diameter coated with TiN. The substrates were polished before coating. A comprehensive characterisation of the coated samples was carried out [8].

Two abrasives were used. The alumina abrasive, which has a nominal particle size of 1 μm was for the non-perforating tests, and the SiC abrasive, which has a nominal particle size of 4 μm was for the perforating tests. Bearing steel balls 25.4 mm in diameter were also supplied.

3.2. Testing

Two different types of testing were required in the interlaboratory exercise. These are perforating tests, where a sequence of craters is produced on the sample of different test durations, the size of the resultant craters measured, and the results analysed to yield wear rates for the steel substrate and the TiN coating. In these tests, the TiN coating is intentionally perforated following the basic method first suggested by Rutherford and Hutchings [9], since updated [6]. These tests were carried out using the SiC abrasive, although some additional non-perforating testing was carried out by two participants using alumina abrasive.

The other type of test was carried out using the alumina abrasive. Here, the test conditions were carefully controlled to ensure that perforation of the coating did not occur. The diameter of the crater was measured and the wear rate of the coating alone calculated.

The procedure given in Appendix A also required a few subsidiary experiments to be carried out with diamond paste to measure the thickness of the coating. They can be carried out with either the fixed ball type of test system, for example as shown in Fig. 4a, or the free ball test system (Fig. 4b).

Both commercially manufactured and “home-built” equipment was used in the interlaboratory exercise (Table 1). Fourteen organisations participated as listed in Appendix B. It should be noted that the order of participants specifically does not correlate with the list of laboratories given in the figures.

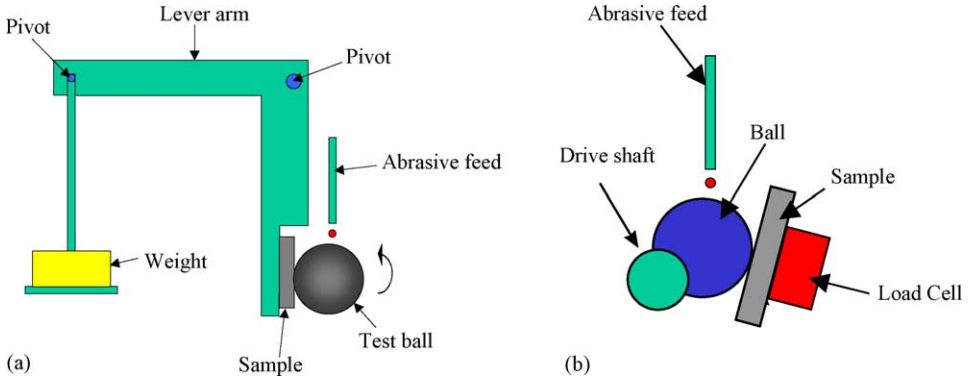

Fig. 4. Main different types of ball cratering systems (a) fixed ball and (b) free ball.

3.3. Results

3.3.1. Perforating tests

Thirteen laboratories returned results for the perforating tests using SiC abrasive. The results are shown in Fig. 5. (Note that some of the results in Figs. 5 and 6 use the *SN* parameter, where *S* is the relative sliding distance and *N* is the normal applied load, and according to the Archard law, the wear volume should be proportional to this parameter for many materials.) Fig. 5a shows the agreement for different laboratories in terms of total volume of the craters, Fig. 5b in terms of the inner carter diameter and Fig. 5c in terms of the

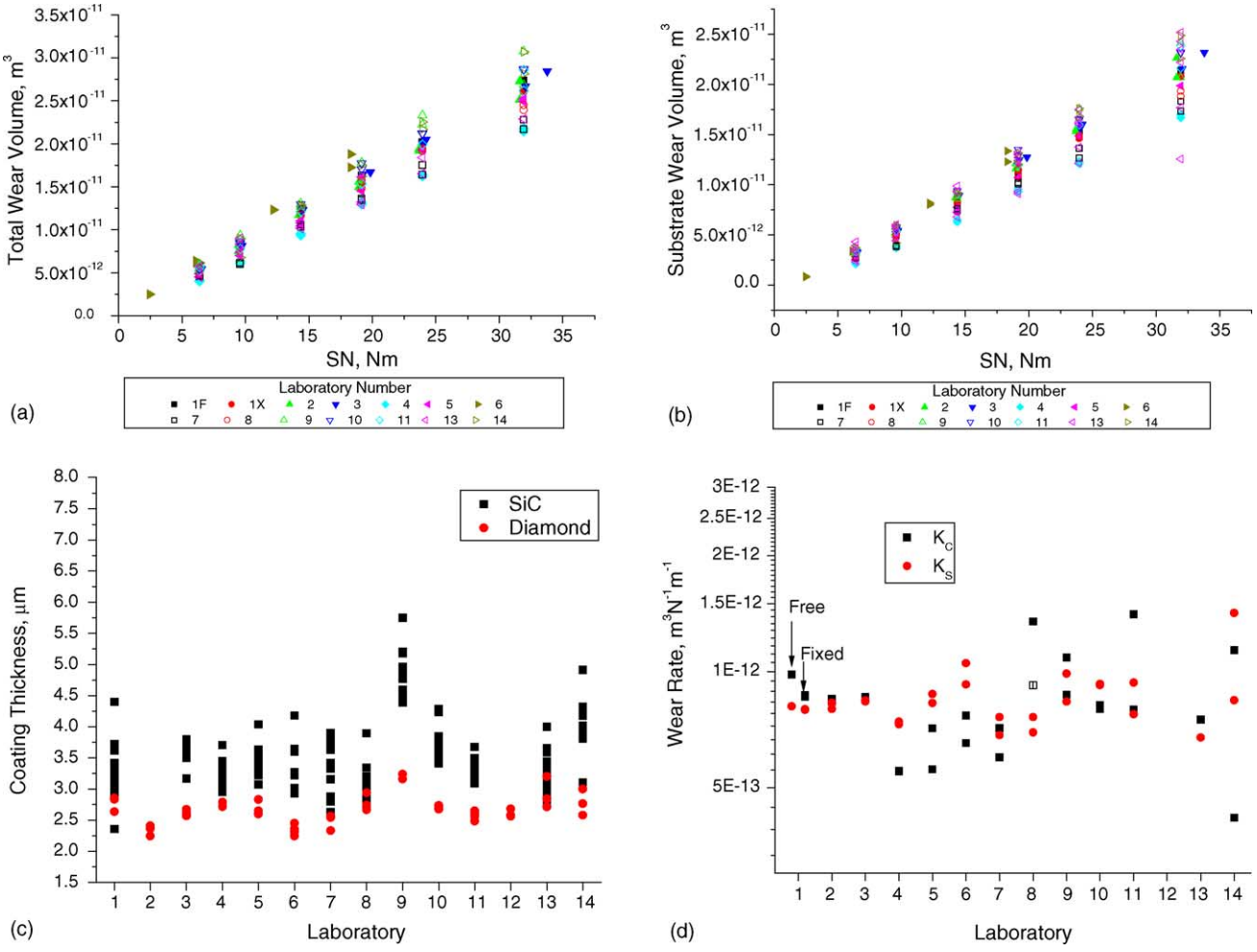

Fig. 5. Interlaboratory exercise results for perorating tests with SiC abrasive (a) total crater volume, (b) substrate crater volume, (c) thickness calculated from crater dimensions for SiC and diamond abrasive and (d) wear rates.

Table 1
Test systems used by participants

Machine	Participant
Plint TE66 (fixed ball)	NPL, CU, Nottingham University, LSGS, Sheffield Hallam University, VITO, TNO, Tohuku University, Southampton University
CSEM instruments calowear (free ball)	Ion Bond
Other free ball	IST, NPL
Other fixed ball	Coimbra, Teer Coating Limited
Gencoa free ball	HEF

thickness calculated from the crater measurements. There is some scatter in the results, but it should be noted that every laboratory had a different sample so that some of the scatter in results may be due to variation in sample thickness from one sample to another. The graph of calculated thickness shows that the thickness calculated from the outer crater diameter is always much larger than that found for the diamond abrasive. This is likely to be due to the observed difficulty in measuring the true diameter of craters made with the relatively large SiC abrasive due to rounding of the edge of the crater and scuffing around the periphery of the crater. Fig. 5c is also suggestive of a systematic variation in the thickness of the samples from laboratory to laboratory, but this may equally be due to systematic variations in measurement method from one laboratory to another.

Fig. 5d gives a comparison between the wear rates calculated by the analysis procedure given in Appendix C. It can be seen that there is some variation in wear rate for the different laboratories, and that with this abrasion, the wear rate for the steel substrate is the same as the wear rate for the TiN coating. It should also be noted that the fixed ball and free ball results for laboratory 1 are very similar.

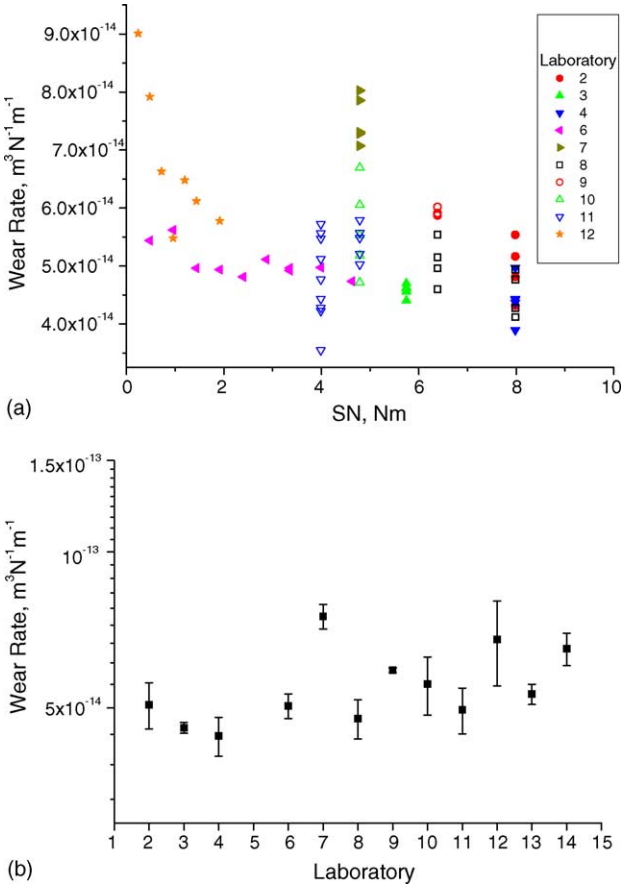

Fig. 6. Variation of coating wear rate in non-perforated alumina tests (a) with SN and (b) comparison of mean and spreads for different laboratories.

3.3.2. *Non-perforating tests*

Fig. 6 gives the results of the non-perforating tests. There seems to be a systematic reduction in wear rate with increasing *SN*, particularly for the two laboratories that carried out tests at low values of *SN*, where the crater size is small. At higher values of *SN*, this systematic variation is obscured by the variation in measurement form one laboratory to another.

3.3.3. *Comparison of results from perforating and non-perforating tests*

Two laboratories carried out tests with 10% alumina abrasive using both the perforating and the non-perforating test procedures. Good agreement was found between the coating wear rate, K_c results for the non-perforating and perforating tests. However, a considerable difference was found between the different laboratories for the substrate wear rate, K_s.

4. Analysis of reproducibility and repeatability

The reproducibility and repeatability of the results were analysed following the procedure laid down in ISO 5725 Part 2 [10]. The results of this analysis are given in Table 2.

For the non-perforating tests for the coating wear rate, K_c the repeatability (within labs) standard deviation, sr is 24% and the reproducibility (between labs) standard deviation, sR is 26%. Again in the perforating tests, for the substrate wear rate, K_s, sr is 7% and sR is 11%. For the non-perforating test, coating wear rate, K_c, sr is 8% and sR is 17%. For the measurement of crater diameter, sr is 2% and sR is 3%.

These values of reproducibility and repeatability are considered to be acceptable for a wear test and are comparable to other wear tests. Thus, the ASTM G99 pin-on-disc sliding wear test quotes a standard deviation of wear scar diameter of 0.27 for *a* value of 2.11, or 12.8% [11] compared with the reproducibility calculated here for *b* of 2.9%, and the ASTM G133 reciprocating test ASTM G133 quotes a standard deviation of wear volume for silicon nitride of 0.189 on a mean value of 0.543 mm^3, or 34.8% compared with the reproducibility calculated here, for example for K_c in the perforating tests of 26% [12] (Table 3).

Table 2
Comparison of perforating and non-perforating tests

Laboratory	Non-perforating, K_c (10^{-14} m^3 N^{-1} m^{-1})	Perforating, K_c (10^{-14} m^3 N^{-1} m^{-1})	Perforating, K_s (10^{-14} m^3 N^{-1} m^{-1})
A	5.5	5	17.80
B	6.5	5.7	288.50

Table 3
Results of analysis of reproducibility and repeatability to ISI 5725 (figures in brackets are percentage of means)

	Perforating tests		Non-perforating tests	
	K_c, 10^{-13} m^3 N^{-1} m^{-1}, N = 13	K_s, 10^{-13} m^3 N^{-1} m^{-1}, N = 13	K_c, 10^{-13} m^3 N^{-1} m^{-1}, N = 12	b, mm, N = 4
Mean	8.00	8.34	5.35	0.561
sr	1.94 (24)	0.57 (7)	0.41 (8)	0.011 (2)
sR	2.09 (26)	0.92 (11)	0.94 (17)	0.016 (3)

N gives number of participants in category.

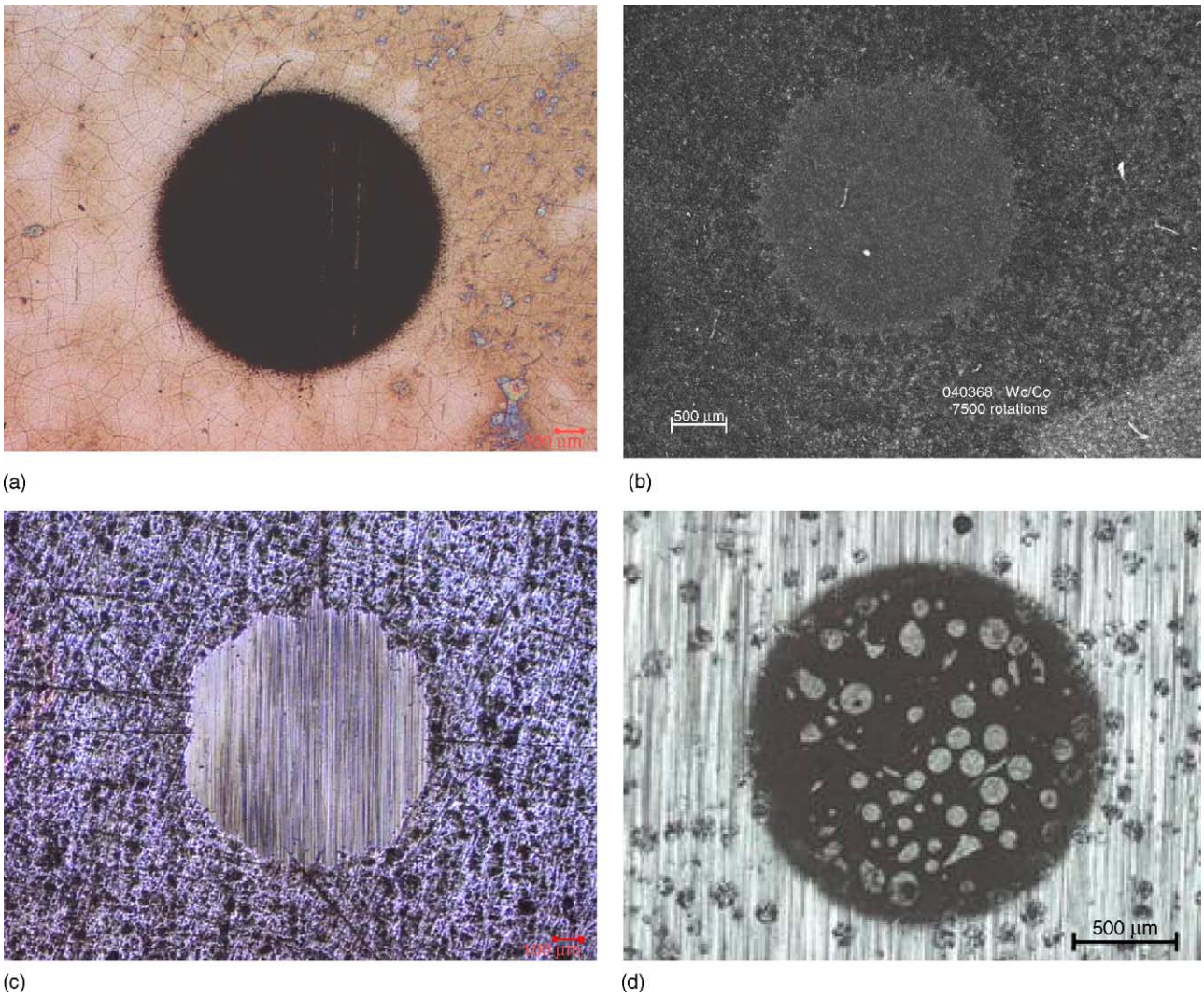

Fig. 7. Examples of micro-scale abrasion test craters on (a) hard chrome plating using SiC abrasive, (b) WC/Co thermally sprayed coating, (c) anodised aluminium and (d) WC/Ni laser cladding.

5. Applicability to different surface engineered surfaces

To assess the applicability of the test technique to different surface engineering processes, a wide range of surfaces with different coatings and surface modifications were tested. The full set of results is given in a separate report with only an indication given here [13].

It was found that a wide range of treatments could be assessed by the micro-scale abrasion technique. This included several other hard wear resistant coatings including CrN and DLC, thick coatings such as thermally sprayed WC/Co and hard chrome, and surface treatments such as anodising. Fig. 7 shows that for all four materials shown reasonably well shaped craters are formed that could be easily measured to obtain a good estimate of wear rate. The reason that the outline of the thermally sprayed WC/Co and the anodised aluminium craters is not as regular as the craters for the chrome plating, the laser cladding and TiN (Fig. 1b) is because these samples are somewhat rougher than the required for the best micro-scale abrasion testing. These results give confidence that although the interlaboratory exercise was carried out specifically on TiN coatings, similar reproducibility and repeatability could be expected for other similar coatings.

6. Conclusions

An interlaboratory exercise was carried out to validate the test procedure that has been used to form the revision to ENV 1071-6, micro-scale abrasion of coatings. The procedure has been revised through the EU project CRATER that finished recently. A new ASTM standard is being drafted that is based on this procedure.

The interlaboratory exercise had 14 participants. It was found that the micro-scale abrasion technique has reasonable reproducibility and repeatability for TiN coatings that compared well with other wear tests.

The technique has been shown to be applicable to the evaluation of the wear performance of a wide range of different surface engineering processes.

Appendix A. Procedure for micro-scale abrasion testing

A.1. Scope

This procedure describes the method for performing wear tests on coated samples by a perforation test, producing a series of craters at increasing time intervals/number of rotations, or by performing a non-perforation test on coated samples.

Table A.1
Mass of abrasive required for 100 cm^3 of water

Abrasive	Size (μm)	Density (g cm^{-3})	Concentration (vol.%)	Mass (g)
SiC	4	3.2	20	80.0
Al_2O_3	1	4.0	10	44.4

A.2. Preparation of samples

Check the sample condition, scar position and direction of ball motion relative to the surface being tested. The samples should be washed to remove all traces of grease. A separate clean steel sample is required for ball surface preparation (running-in procedure). Any steel with hardness between 200 and 900 HV should be suitable.

A.3. Preparation of abrasive slurry

Leave deionized water in an open container in laboratory for at least 1 h (for CO_2 absorption).

Add deionized water to the appropriate weighed quantity of abrasive powder (Table 1 lists the mass of abrasive for each 100 cm^3 of water).

Mix thoroughly to produce a uniform suspension of particles with a concentration of 20 vol.% for the SiC slurry and 10 vol.% for the alumina slurry (Table A.1).

A.4. Preparation of the ball surface (run-in procedure)

The ball should be washed to remove all traces of grease. A steel sample (for the run-in procedure) should be mounted in the apparatus. The motor speed should be adjusted to give the correct value for the rotational speed of the ball (in the range 0.1 ± 0.01 m s^{-1} of sliding speed), and the normal loading between the ball and sample adjusted to give the correct value of 0.2 ± 0.2.

The method of feeding slurry to the contact point can vary between instruments. It may be via a pump or applied manually by the operator. Care must be taken to ensure that the slurry remains well mixed. The feed rate shall be sufficient that the contact between the ball and sample is always well wetted by the slurry. The slurry shall be used once only and not re-circulated.

Load the sample and ball together having ensured that the sample and ball are pre-wetted by the slurry, and then start the motor and timer or counter. Typically 5 min running time, or between 300 and 400 revolutions should be used for the running-in procedure.

Change the orientation of the ball and repeat from stage 4.6.6 until more than 5 run-in tracks are produced randomly on the ball. The ball is now conditioned for further use. Typically such a ball can be used for at least 10 samples (>60 tests).

Table A.2
Test conditions for perforating tests (found to be appropriate for TiN on steel; other test conditions may be appropriate for other coatings)

Load (N)	0.2 ± 0.02
Speed ($m\,s^{-1}$)	0.1 ± 0.01
Ball material	As supplied
Ball diameter (mm)	25.4
Ball finish	Conditioned using run-in procedure
Abrasive material	SiC as supplied
Fluid carrier	Water
Feed rate	Keep wet
Abrasive concentration (vol.%)	20
Test duration (approximate number of ball revolutions)	400, 600, 900, 1200, 1500, 2000

Table A.3
Test conditions for non-perforating tests found to be appropriate for TiN on steel, other conditions may be appropriate for other materials

Load (N)	0.2 ± 0.02
Speed ($m\,s^{-1}$)	0.1 ± 0.01
Ball material	As supplied
Ball diameter (mm)	25.4
Ball finish	Conditioned using run-in procedure
Abrasive material	Al_2O_3 as supplied
Fluid carrier	Water
Feed rate	Keep wet
Abrasive concentration (vol.%)	10
Test duration (approximate number of revolutions)	500, but perforation must not occur

A.5. Test method for perforating tests

Mount the test sample in the apparatus so that the centre of the crater that will be formed will be at least 3.5 mm away from any previous crater. Confirm that the motor speed is the correct value to give the required rotational speed of the ball. And confirm that the correct normal load is achieved. Use 20% SiC slurry prepared as described earlier. The slurry feed shall be adjusted so that the contact between the ball and sample is always well wetted by the slurry. The slurry shall not be re-circulated (Table A.2).

After loading the sample and ball together having ensured that the sample and ball are pre-wetted by the slurry the motor and timer or counter shall be started. When the set time interval or number of revolutions is completed, switch off the motor, stop the slurry feed and remove the ball or load.

Clean the ball and the test sample using a fine jet of water and dry the sample with tissue. Repeat at a new position of the sample with different time interval or number of revolutions until the series of tests for the same condition is completed. For the standard condition, six tests with different durations should be performed. The test durations given in Table 2 are recommended. For each test, use a new orientation of ball relative to the sample. Normally two complete series of six tests should be carried out.

A.6. Test method for non-perforating tests

The test method is the same as for perforating tests, but the test duration is adjusted to ensure that perforation of the coating does not occur. Some trial and error may be required to achieve this, but preliminary tests have shown that the test duration given in Table A.3 is appropriate for TiN coatings on steel

A.7. Measurement of coating thickness

To measure the thickness of the coating, a fine metallographic diamond paste or spray (1 μm abrasive size or less) should be used to create craters at three positions well spaced over the surface of the sample. The outer crater diameter should be about 0.7 mm. The inner and outer crater diameters, a and b, respectively, measured from these craters (see Fig. A.1) are used to calculate a coating thickness at each position. The three values of coating thickness are used to calculate an average coating thickness for the sample. The formula for the coating thickness, t is:

$$t = R(\alpha - \beta).$$

where R is the radius of the ball and

$$\alpha = \left(1 - \frac{a^2}{4R^2}\right)^{1/2} \quad \text{and} \quad \beta = \left(1 - \frac{b^2}{4R^2}\right)^{1/2}.$$

A.8. Measuring method

The dimensions of the wear craters in two directions (parallel, // and perpendicular, ⊥ to the direction of ball motion) of both the inner a and outer b crater diameters are measured (see Fig. A.1). For the non-perforation tests, measure the crater diameters b in the two directions parallel, // and perpendicular, ⊥.

A.9. Data report and analysis

Report details of the test system that was used in the experiment. Note any specific test conditions that were used

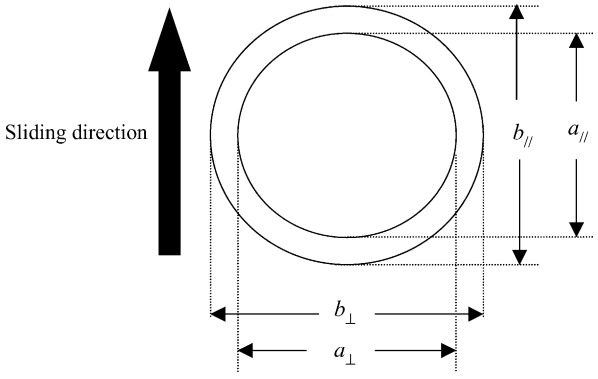

Fig. A.1. Measurement on wear scar.

such as the sample inclination, shaft diameter and groove dimensions in a free ball machine. Report the results of the thickness measurements calculating the coating thickness using the formula given earlier.

For the perforating tests report sample number, scar position, test conditions (type of micro-abrasion test (fixed or free), ball diameter, abrasive material, fluid carrier, abrasive concentration, rotation speed, normal load and number of revolutions) and measured diameters $a_{//}$, $b_{//}$, $a_{\perp}$ and $b_{\perp}$ for each crater. Use the analysis procedure in Appendix C to calculate wear rate results.

For the non-perforating tests report all test results the wear rate can be calculated from

$$K_c = \frac{1}{SN}\frac{\pi a^4}{64R}$$

Appendix B. List of participants

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(2) HEF R&D, France
(3) IonBond Ltd., UK
(4) Laboratoire de Science et Genie des Surfaces (LSGS), Ecole des Mines, Nancy, France
(5) National Physical Laboratory, UK
(6) Nottingham University, UK
(7) Sheffield Hallam University, UK
(8) Teer Coatings Ltd., UK
(9) TNO, The Netherlands
(10) Tohoku University, Japan
(11) Universidade de Coimbra, Portugal
(12) University of Cambridge, UK
(13) University of Southampton, UK
(14) VITO Belgium

Appendix C. Analysis method for perforating tests

For each crater, calculate the volume of wear in the coating, V_c and the volume of wear in the substrate, V_s from the values of a and t. The formulae below should be used for practical purposes and these approximate expressions are sufficiently accurate.

$$V_c = \frac{\pi t}{4}(a^2 + 4Rt)$$

$$V_s = \frac{\pi a^4}{64R}$$

Plot a graph of SN/V_c against V_s/V_c for all the craters (S is the total distance of relative movement and N is the normal applied load. The data points should lie close to a straight line. Any outlying points may be disregarded or repeat experiments performed before subsequent data analysis. Apply linear regression (least squares method) to determine the line of best fit to the data points. Obtain the coating wear coefficient, κ_c from the intercept and calculate the substrate wear rate coefficient, κ_s from the slope, as indicated by the equation:

$$\frac{SN}{V_c} = \frac{1}{\kappa_s}\frac{V_s}{V_c} + \frac{1}{\kappa_c}.$$

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