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Short note on improved integration of mechanical testing in predictive wear models

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Abstract

In this work, a new global increment nano-fretting wear model based on the effective indenter concept has been used and the results were compared with experimental data. A series of DLC coatings with varied mechanical properties was deposited using industrial scale PECVD system and characterised on a low-drift nanomechanical test platform (NanoTest Vantage). 4500 cycle nano-scale fretting measurements have been performed in order to examine the tribological properties of the coatings. A physical analysis of the nanoindentation test enabled the true coating Young's Modulus (E) and the coating yield strength (Y) to be determined. In comparison to the hardness (H) this is the basis for a more generic understanding of the mechanical coating behavior. This allowed direct examination of the influence of the variation of Y/E in the coatings on the observed nano-fretting wear, with the coating with highest Y/E showing significantly improved resistance to nano-fretting wear. A preliminary evaluation of the stress field evolution during the test and the extraction of wear and fretting parameters provides the opportunity to discuss the effects possibly being dominant within the nano-scale tribo-tests.

1. Introduction

Improved integration of measurement data obtained from mechanical testing is required to provide reliable inputs for predictive wear models. Due to complex nature of tribological processes a large number of theoretical wear models have been proposed. It was estimated by Meng et al. [1] that 183 equations with more than 300 material and mechanical parameters describing the wear process can be listed. These models often have a theoretical-experimental character. Hence, in order to estimate the wear coefficients a series of tests have to be completed. Moreover, models prediction potential remains relatively low as they are usually limited to a specific tribo-system and it is hardly possible to employ them to any other practical situation. Nevertheless, the tribologists make every effort to find a universal approach to estimate engineering components lifetime by developing more advanced wear models.

Early wear models originated from empirical equations developed directly from tribological experiments. In 70's and 80's of the 20th century, wear models started being elaborated on the basis of mechanical contact. Many of them took into account the real contact area and mechanical properties of the materials like Young's modulus and hardness. The most recognised wear model from that period was introduced by Archard [2]. This model was proposed well in advance to other laws of contact mechanics and was derived by Archard from an equation previously given by Holm [3], in which a dimensionless coefficient K was introduced to provide the conformity of the formula with experimental results. The K coefficient was interpreted by Archard as a probability to form a wear particle by the asperities of the interacting solid bodies, however other authors propose different interpretations of this coefficient [4-6].

Fretting wear is a specific surface destruction process, where the reciprocating sliding motion with relatively small amplitude is responsible for the debris formation and loss of material by interfacial shear work. Nano-fretting data have been reported by several research groups carrying out experiments on various, commercially available and purpose-built equipment. Performing the tests at this scale with well-defined spherical probes as model single asperity contacts simplifies the contact geometry and provides a route for deeper understanding of the fundamental phenomena. Varenberg et al. studied partial and gross slip fretting behaviour of 3.1 μm diameter scanning probe microscopy probes tested against Si wafers [7]. Nano-fretting behaviour of monocrystalline silicon for potential application in MEMS devices operating in vacuum conditions was studied by Yu et al. using AFM tips [8-9]. The energy ratio related to the transition from partial to gross slip regime was measured and compared to the same energy ratio observed in classic macroscale fretting. The authors looked also at 2 nm thick DLC coatings deposited on Si(100) and carried out tests against SiO2 microspheres under vacuum and air conditions [10]. They found that DLC coating reduced significantly adhesion and friction force in air conditions comparing to Si(100) substrate. Wilson et al. focused on C and Cr doped amorphous C films and carried out small scale fretting experiments using a modified nanoindenter (NanoTest) with 300 μm diameter ruby tip under 10-200 mN applied load and 2-14 μm displacement amplitude [11-13]. The authors identified two distinct fretting wear regimes, with classic W-shaped wear scar under low oscillation amplitude and full U-shaped wear scar at larger amplitudes.

Beake at al. recently used a modified nanoindenter platform for reciprocating testing under milli-Newton range constant or ramped applied normal loads. In [14] nanoindentation, nano-scratch and nano-fretting tests were performed and compared on highly polished Si(100) using a 4.6μm sphero-conical diamond indenter to assess the influence of tangential loading on the deformation bheaviour. In [15,16] nano-fretting of 5, 20 and 80 nm thickness ta-C films deposited on Si(100) was investigated using spherical indenters, where it was found that fretting wear occurred at significantly lower contact pressure than is required for plastic deformation and phase transformation in indentation and scratch experiments. Finally in [17] nano-fretting experiments were carried out on biomedical grade Ti6Al4V, 316L stainless steel and CoCr alloy samples using 3.7 μm sphero-conical diamond indenter.

Analytical tools for nanoscale materials testing have rapidly developed over the last three decades. Improved resolution and efficiency of existing techniques enabled development of new experimental tools including nano-fretting testing and posing new challenges for predictive models. In this work we have characterised the mechanical properties of three different DLC coatings on M2 steel by nanoindentation and performed 4500 cycle nano-fretting tests on them. We report on the first attempt of improved integration of measurement data obtained from mechanical testing providing reliable inputs to nano-fretting wear model and also provide an initial comparison between the experimental nano-fretting data and the wear prediction from the model for the coating with the highest Y/E ratio.

2. Experimental

2.1. Materials

A series of three DLC coatings (denoted A, B and C) with varied mechanical properties was deposited on M2 grade steel substrates for this study. Coatings A and B were deposited using industrial scale PECVD Flexicoat 850 system (Hauzer Techno Coating, the Netherlands) in the Advanced Coatings Design Laboratory in School of Mechanical Engineering at the University of Leeds while coating C was a commercial coating, Balinit C Star, obtained from Balzers company. Coatings A and B were deposited with chromium and graded tungsten carbide interlayers in order to enhance adhesion between the DLC coating and the substrate. The Cr layer was deposited using magnetron sputtering, while the WC layer was deposited using magnetron sputtering with the gradual introduction of Acetylene gas to the complete PACVD stage, thus creating a functional gradient layer in one continuous deposition process. Additionally, coating B was doped with silicon using hexamethyldisiloxane (HDMSO) precursor. According to Balzers specification, Balinit C Star is applied in a single-pass vacuum process at temperatures between 180 and 350°C, resulting in homogeneous coating structure. Thickness of the coatings was assessed using Calotester (Tribotechnic, France) employing abrasion ball cratering testing method. Surface roughness was measured using two-dimensional contacting profilometry (Form Talysurf series, Taylor-Hobson, UK). Surface roughness data was analysed to the least square line, with Gaussian filter, 0.25mm upper cut-off and bandwidth 100÷1.

Table 1: DLC coatings.

Coating	Coating type	Coating thickness (μm)	Roughness Ra (nm)
Coating A	Cr + W-C:H + DLC	2.9	15.3
Coating B	Cr + CrW + W-C:H + Si-DLC	2.8	9.3
Coating C	CrN + a-C:H:W	2.0	17.8

2.2. Nanoindentation and nano-fretting experiments

Nanoindentation and small scale fretting experiments were performed using the NanoTest Vantage (from Micro Materials Ltd., Wrexham, UK) using the nanoindentation and nano-fretting modules. Nanoindentation experiments were performed in order to characterise the hardness and Young's modulus of the coatings. The experiments were performed on the three coatings with a sharp Berkovich indenter to peak loads of 1, 5, 10, 20, 30, 40, 50…100 mN with 10 repeats for each load. The loading time was 20s with a hold of 5s before unloading in 20s. Data were corrected for thermal drift using hold periods of 60s prior to the load ramp and after 90% unloading. Data from the nanoindentation experiments were also exported from the NanoTest software and analysed using Oliver and Pharr for Coatings software (SIO, Ummanz). This software allows determination of the coating-only Young's modulus and yield stress from conventional nanoindentation data. Full details of the operation of the software have been described elsewhere, as discussed in section 2.3.

Full details of the experimental hardware used in the small scale fretting tests have been published in previous works [11-15, 18]. For the fretting experiments reported in the current study a 5 µm diamond probe was used, the fretting parameters chosen were 100 mN load, 13.4 µm track length and 5 Hz frequency. Experiments ran for 15 minutes at peak load so that each sample was subjected to 4500 cycles. During the experiments load, depth and tangential force were recorded in order to assess the damage. Friction coefficients during the fretting tests were determined from friction loops by the method of Burris and Sawyer to eliminate any potential transducer misalignment issues [19]. The 100 mN load was applied over 20 s at the start of the experiment, there was then a 10 s hold before the fretting started to allow the initial on load depth to be recorded. At the end of fretting there was again a 10 s pause to record the final on load depth. The load was then removed over 20 s with a 300 s pause at 90 % unloading to assess any thermal drift which may have affected the measurement. After unloading a final 10 s with the probe held at the surface was used to assess the residual depth of the fretting scar. During analysis the data were corrected for any thermal drift during the experiment. An example of a typical loading profile is shown in Figure 1. Three repeat 4500 cycle tests were performed on each coating.

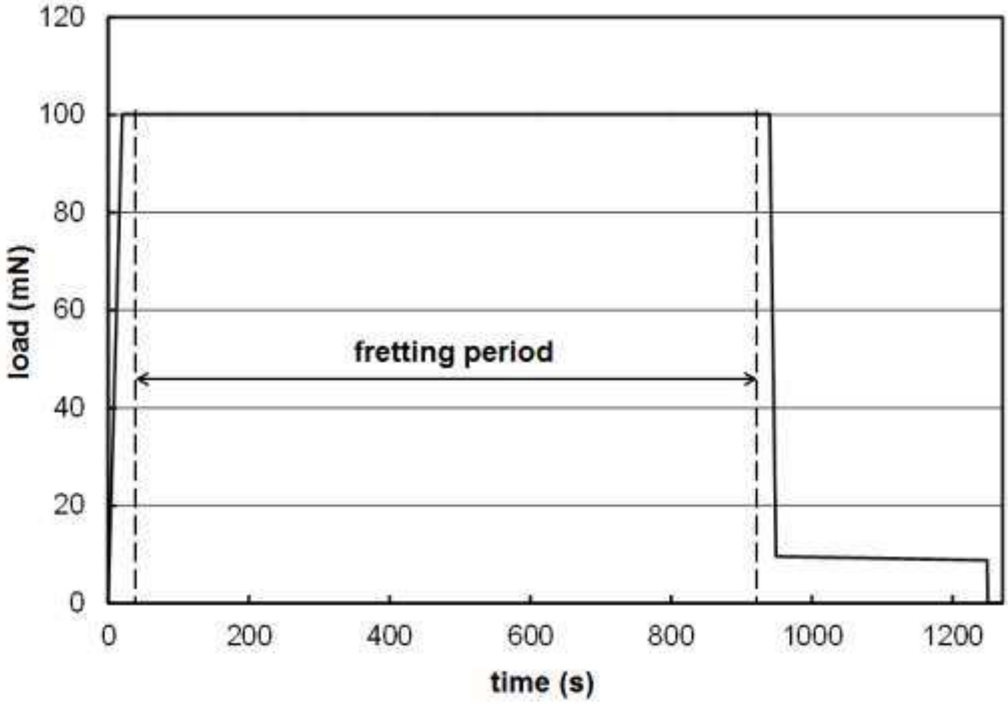

Fig. 1. Typical loading profile for a fretting experiment.

2.3. Wear model details

A predictive (physical) description of wear tests requires the combination of a variety of scientific fields and concepts of material science. Due to lack of space within this short note we will not elaborate all the theoretical material here, but refer to other papers instead [20,21]. The following issues need to be covered by the theoretical apparatus necessary for a proper wear or fretting model:

- first principle based interatomic potential description of mechanical material behavior [22];
- the effective indenter concept [23,24];
- the extension of the Oliver and Pharr method to analyze nanoindentation data to layered materials and time dependent mechanical behavior [25-27];
- the physical scratch and wear test and its analysis [28,29].

The main difference to more “classical” ways to describe wear effects [e.g. 30,31] might be seen in its successive or even hierarchic built-up of the model from first, but effective principles to phenomenological parameters and from static or quasi static measurements to dynamic tests (Fig. 2).

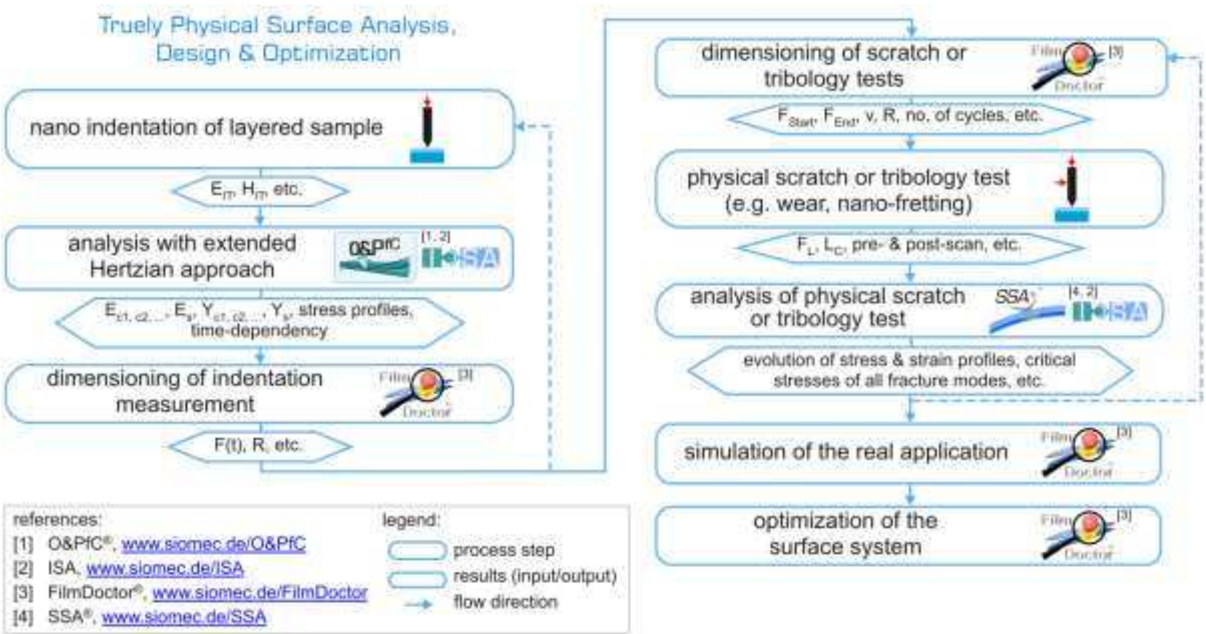

Fig. 2. A flow chart of the procedure of mechanical characterization and optimization of arbitrary structured surfaces with respect to wear and fretting.

Some of these aspects shall be considered more closely within this paper, whereby we pay explicit attention to the coupling of the deformation field (mainly the stress tensor) into the decomposition behavior of the material determining its wear and fretting properties.

A more general simulation describing our nano-fretting or nano-wear experiments cannot be performed via connecting the wear effect by a simple k_d-value (simple Archard’s law) to the deformation or stress field. Instead, in the most simple and linear case, the tribo-effect is a tensor (often of even operational character), coupling with wear-moduli to every deformation field component in a fully covariant manner. More correctly any tribo-process could be linearly generalized like:

$$\mathrm{tribo-effect}_{ij} = k^{\sigma}{}_{ijkl}\ \sigma^{kl} + k^{\varepsilon}{}_{ijkl}\ \varepsilon^{kl} + k^{u}{}_{ijkl}\ u^k u^l + \sum_{n=1}^{N} k^{S_n} \delta_{ij} S_n \tag{1}$$

Here we used the following denotations: $k^{xx}{}_{ijkl}$-tensors are tensors coupling to the various field values or tensors like stress σ^{kl}, strain ε^{kl}, displacement-vector u^i or scalar values S_n, like free or distortion energy strain work etc.. The symbol δ_{ij} is the Kroenecker symbol. In most cases, wear for instance, it should be sufficient to consider only the stresses:

$$\mathrm{tribo-effect}_{ij} \equiv w_{ij} = k_{ijkl}\ \sigma^{kl} \tag{2}$$

Wherefrom the scalar wear-depth h_w has to be evaluated via:

$$h_w = w_{ij}\ n^i\ n^j \tag{3}$$

With n^i denoting the surface normal unit vector.

As we can see, the Archard's law given with a scalar wear coefficient k_d by the simple relation $h_w=k_d * \sigma^{33}$ is nothing but a rather dramatic simplification of equation (2). Within this paper we will make use of an operational wear law taking into account the von Mises stress maximum σ_M, but also its distance to the surface contact centre $|\vec{r}|$. It is given as

$$w_{ij} = \delta_{ij} \left(k_{dM} \sigma_M \cdot e^{-\lambda_k \vec{r} \cdot \vec{r}} \right) \tag{4}$$

with λ_k being yet another parameter in addition to the linear k_d. A more comprehensive evaluation and discussion was given in [32].

3. Results

3.1. Nanoindentation

Nanoindentation revealed clear differences in mechanical behaviour between the different DLC coatings confirming their suitability as model samples for the study of Y/E on fretting wear. As an illustration the results from tests to 20 mN peak load are shown in Table 2. There is a clear difference in H/E_r (and Y/E) with Coating A having the highest H/E_r ratio and Coating C the lowest. The variation in H/E_r ratio with increasing indentation depth is shown in Figure 3.

Table 2. Nanoindentation testing to 20 mN

	H (GPa)	E_r (GPa)	h_c (nm)	H/E_r
Coating A	23.4 ± 1.1	194.9 ± 8.9	159 ± 5	0.120
Coating B	16.2 ± 0.6	143.0 ± 3.1	196 ± 4.3	0.113
Coating C	11.5 ± 0.9	146.1 ± 5.6	238 ± 10	0.079

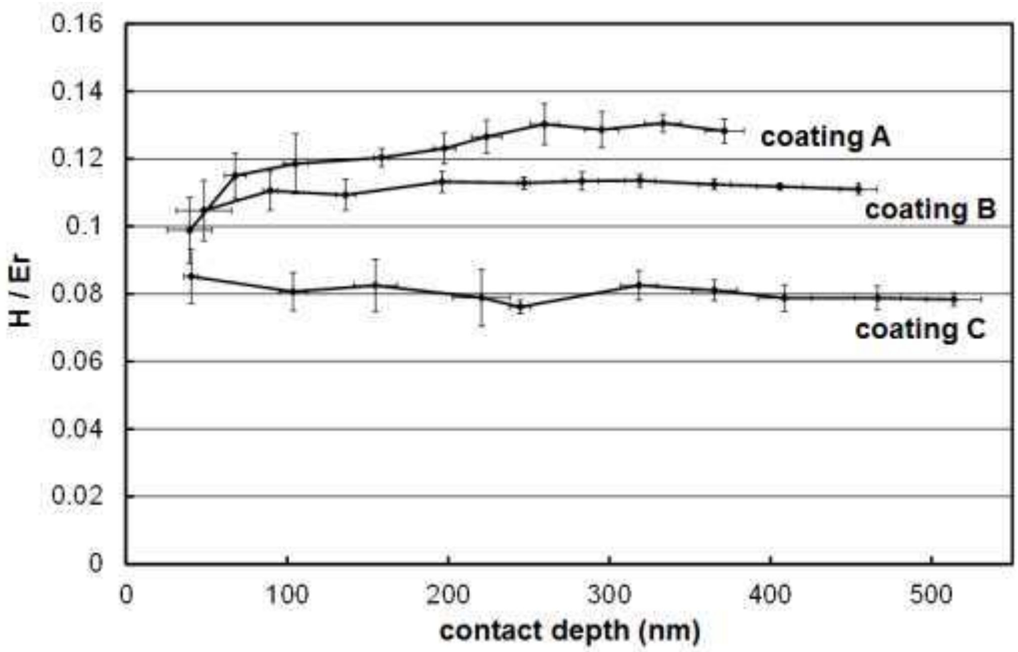

Fig. 3. Variation in H/E_r with indentation contact depth

3.2. Nano-fretting

The evolution of on-load probe depth and the friction force during the test are illustrated in Figure 4 for Coating A. In this test the friction initially increased then reaches a minimum after 1500 cycles before gradually increasing to the end of the test, though the friction coefficient remains at ~0.15 throughout. The rate of nano-fretting wear is not constant through the 4500 cycle test, decreasing as the test progresses (Figures 4 and 5). This is particularly clear for Coating B whose wear rate decreases significantly beyond ~800 fretting cycles in all the tests. Although the initial wear rate on Coatings B and C is similar the final wear depth (Table 3) is greater on Coating C as the reduction in wear is less.

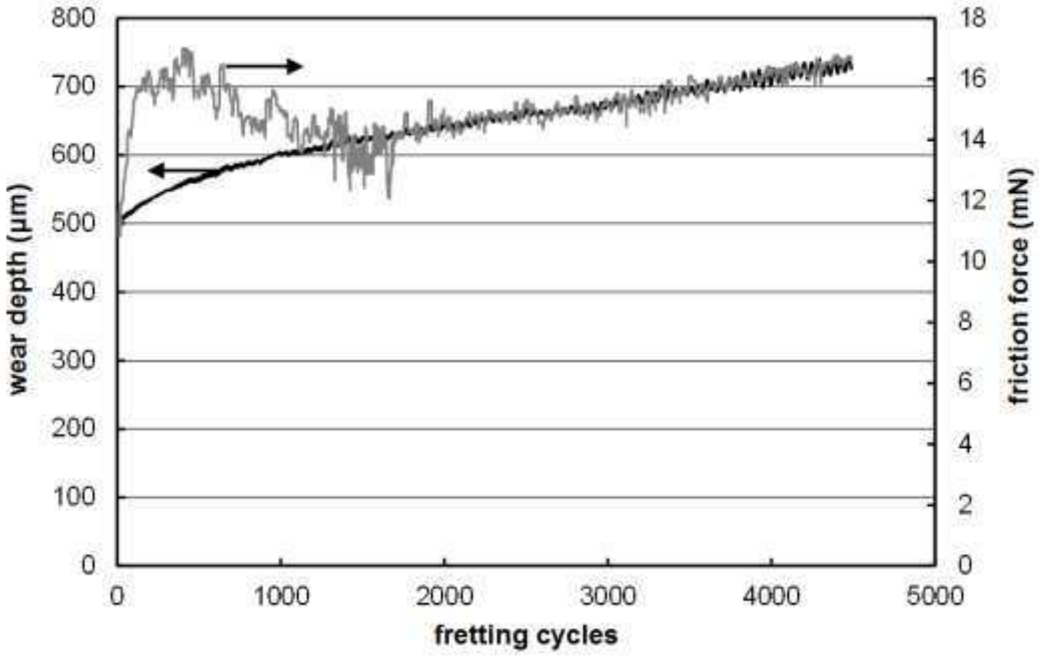

Fig. 4. Coating A, 100 mN, 15 min fretting, R = 5 μm probe, 5 Hz.

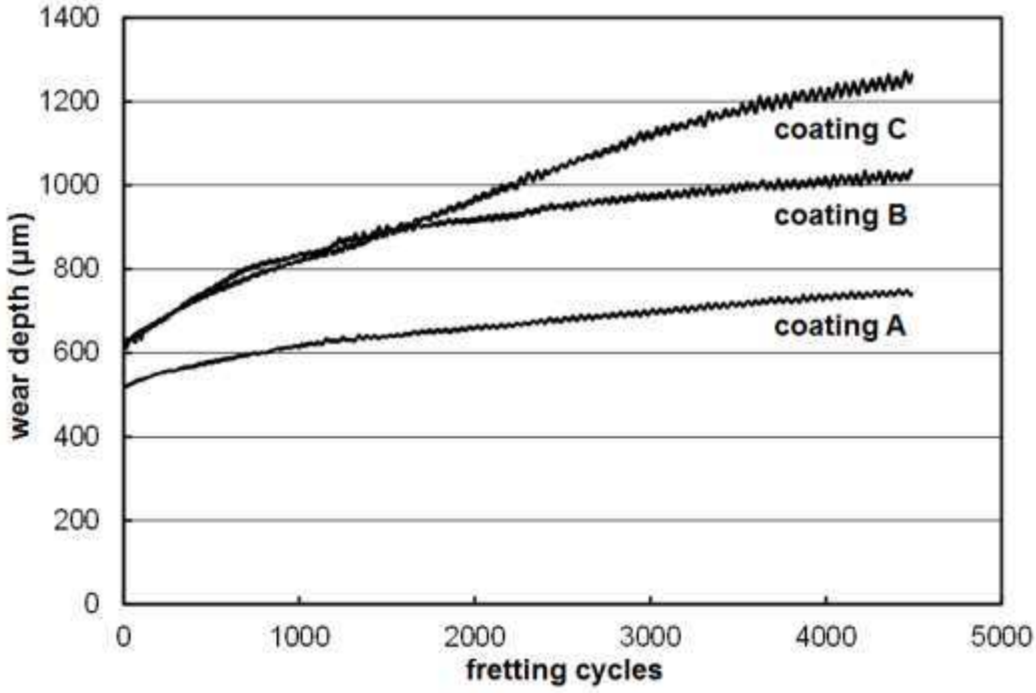

Fig. 5. Nano-fretting wear depth comparison for coatings A, B and C; 100mN, 15 min fretting, R = 5 μm probe, 5 Hz.

Table 3: Fretting results.

Coating	Initial on-load depth (nm)	Post fretting on-load depth (nm)	On-load depth increase (nm)	Residual depth (nm)
Coating A	384 ± 5	685 ± 31	301 ± 26	288 ± 10
Coating B	452 ± 3	1012 ± 62	560 ± 64	608 ± 113
Coating C	435 ± 23	1248 ± 42	813 ± 28	752 ± 154

Results from fretting experiments on the 3 coatings are shown in Table 3, with on-load depth data corrected for the compliance of the fretting stage. The data shows the mean value with standard deviation from three experiments in each case, indicating a clear ranking of the coatings with Coating A experiencing significantly less wear than the other two coatings.

The coatings differ in their friction evolution during the fretting tests. The mean initial and final friction forces are summarised in Table 4. After initial variability the friction force on Coating B remains constant through the duration of the test and on Coating C it increases continuously through the test. Coating A shows more complex dependence, with a clear minimum in friction after 1500 s (as in Figure 4) before rising to values slightly higher than the initial friction.

Table 4. Friction evolution in fretting

	Initial friction coefficient	Final friction coefficient
Coating A	0.11	0.16

Coating B	0.25	0.28
Coating C	0.23	0.33

3.3.Wear model

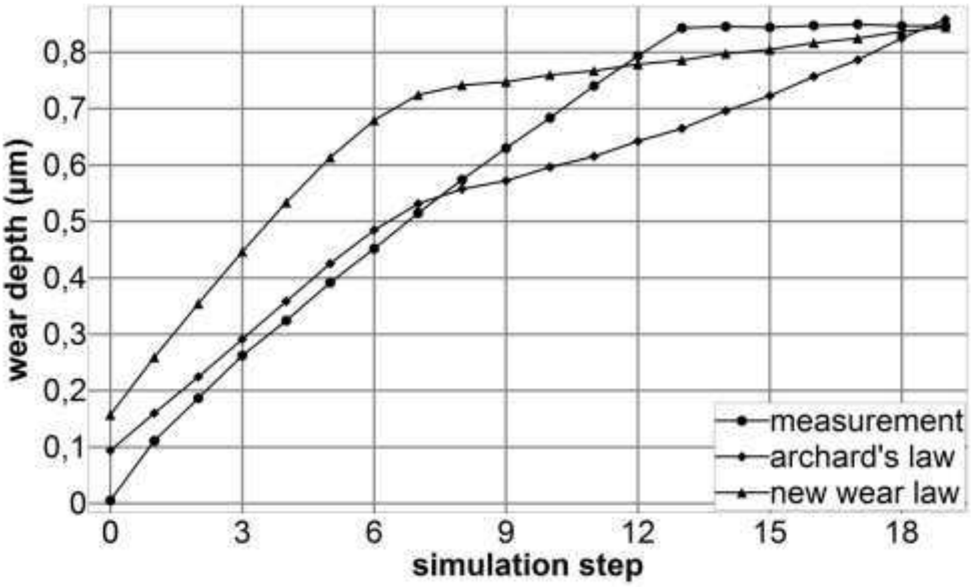

Fig. 6. Experimental nano-fretting data from the start of a test at 200 mN on Coating A and simulated data from wear models

An example of the simulation of experimental nano-fretting data is shown in Figure 6. A clearly non-constant wear rate in the initial stages was observed in the experiments and in both the models. The evaluation ("new wear law" in Figure 6) checks whether Y is exceeded anywhere within the compound (in case of layers it checks substrate and all layers). If this is the case the effective indenter concept is been used in order to account for any plasticity possible having occurred. Taking the plastic surface shape change into account the contact situation is evaluated again (and again…) until Y is not exceeded anywhere.

4. Discussion

The nanoindentation data (Table 2 and Figure 3) clearly show marked differences between the coatings. Data at different peak loads contain differing elastic contribution from the M2 substrate. To obtain accurate coating-only values of E for input to the simulations, it is necessary to remove this substrate component which exists even when indenting to 1/10 of the coating thickness. This has been done by exporting the NanoTest data to the Oliver and Pharr for Coatings software. This yields values of Young's modulus of 227 GPa, 151 GPa and 142 GPa for Coatings A, B and C respectively. As a cross-check, data from across the load range was used in an ISO14577-4 [33] approach to equate the coating-only elastic modulus with the extrapolated value at zero depth. There was very good agreement between the two approaches. The analytical treatment in the Oliver and Pharr for Coatings software has the advantage in requiring acquisition of less experimental data.

The fretting tests reported here were in the gross-slip condition. Observed differences in resistance to nano-fretting wear were correlated with differences in the Coatings Y/E (and H/E_r see Figure 2) ratio. This is consistent with observations at the macro-scale by Leyland and Matthews that wear resistance often correlates better with H/E than H alone [34]. The observed frictional behaviour on repetitive sliding is explained by changes to the ploughing component of friction and smoothing of asperities, both of which alter the contact area and therefore can influence the friction. In a repetitive nano-scratch test the initial friction force typically decreases due to smoothing out surface asperities. Such initial decreases are occasionally seen in the nano-fretting friction. As the fretting test progresses the increasing friction due to increasing contact area due to wear and decreasing contact pressure is observed. The increase in friction through the test is much greater (see Table 4) on Coating C reflecting the much greater wear on this sample. In general higher wear rate was associated with increasing friction, and a transition to lower wear rate more steady friction. Studies of the relative importance of yield stress and microstructure on the evolution of friction and wear of metallic materials during micro-scale repetitive low-pass sliding have concluded that yield stress plays the dominant role on the evolution of friction as the friction was almost independent of the grain size but decreased with increasing hardness [35-37]. Non-constant rate of sliding wear has been observed in repetitive micro-scratch testing of bulk metallic samples such as Cu where the large plastic strains associated with sliding contact can produce a surface hardening effect [36] or surface softening [37]. The lower initial friction on Coating A is consistent with its lower hardness and reduced ploughing contribution.

In the example shown here (Figure 6) we observe the interesting fact, that the classical Archard's law almost perfectly describes the first part of the nano-fretting test, while the new wear law (equation (4)) better fits the behaviour of the later part. As the classical Archard's law only considers the normal surface stress while the new wear law takes into account the complete stress field's deviatoric parts put together in the von Mises stress the following hypothesis might be suggested: during the running in or asperity dominated beginning of the nano-fretting test the wear in fact is mostly determined by surface stress information which is rather perfectly been mirrored by the classical Archard's law. The somewhat later wear however, seems to depend more on shearing processes and shearing stresses underneath the contact zone, which favours the new wear law, because it takes not only these stresses into account, but also considers its distance to the surface. A deeper discussion of these effects on more examples will be given elsewhere.

5. Conclusions

The integrated experimental test configuration described (nanoindentation and nano-fretting with friction measurement) has effectively characterised the mechanical properties and nano-scale tribological behaviour of a series of DLC coatings deposited on M2 steel for anti-wear applications. A physical analysis of the nanoindentation test data was performed enabling the true coating Young's Modulus (E) and the coating yield strength (Y) to be determined. Good agreement was found between the ISO14577 and the above method to determine coating-only modulus. This allowed direct examination of the influence of the variation of Y/E in the coatings on the observed nano-fretting wear, with the coating with highest Y/E showing significantly improved resistance to nano-fretting wear. In general high wear rate was associated with increasing friction. The model introduced here has shown initial promise in the analysis of nano-fretting tribological experiments, reproducing features of the non-linearity in the wear process, and can be used to forward simulate such tests and give hints for better component life-time predictions.

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