Micro Materials
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APPLICATION NOTE

Nano-impact testing of DLC coatings for automotive engine applications

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A rapid reliable test to streamline coating development for improved impact/fatigue resistance.

Introduction

Friction reduction in automotive engines is important to reduce fuel consumption and thus meet environmental and legislative requirements. Diamond-like carbon (DLC) coatings are being increasingly used on many components in the power train (e.g. tappets, pistons, piston rings, fuel injectors..) [1]. Although they work well in fully lubricated conditions it is important to optimise them for components operating under mixed or boundary lubrication. Under boundary conditions the surface is critical in achieving lower friction and wear. Current power train trends such as those towards downsizing, turbocharging, low lubricant viscosity and “start-stop” will increase the number of components operating in boundary/mixed regime. However, it is well known that DLC coatings commonly display poor durability under severe loading conditions [2].

To optimise the performance of DLC coatings operating in demanding contact loading conditions it is useful to assess their mechanical behaviour under more relevant loading conditions than solely in nanoindentation. Films that show higher dynamic toughness should combine (1) a high load threshold for the initiation of cracking (2) mechanisms to minimise and retard crack propagation – i.e. it is necessary to display a certain degree of damage tolerance. A repetitive nanomechanical test – the nano-impact test – has been used to assess film toughness and damage tolerance under dynamic loading. The test measures the degradation of surface engineered materials from repeated localized stresses. The repetitive contacts in the nano-impact test are actual impact events where the probe leaves the surface between each subsequent impact which occurs at the same location on the sample surface. Although nanoindentation can be used to assess toughness as a measure of resistance to crack initiation and overload failure it is not possible to monitor crack propagation under repetitive, oscillating loading conditions in the conventional quasi-static nanoindentation test and for this the dynamic nano-impact test is preferred.

In this Application Note, we have investigated the performance of three DLC coatings on tool steel varying in their composition and mechanical properties.

The Nano-impact technique

The patented nano-impact test is a nanomechanical test technique involving high strain rate contact [3]. The test extends the range of nanomechanical test techniques to much higher strain rates (typically ~10^2-10^3 s^{-1}) than are possible in standard quasi-static nanoindentation (~10^{-2}-10^{-4} s^{-1}). This high strain rate contact allows much closer simulation of the performance of coatings systems or bulk materials under highly loaded intermittent contact and the evolution of wear under these conditions. The progression of surface damage is followed using the depth-sensing capability of the NanoTest. This technique can be used to evaluate: (i) Erosion resistance (ii) Cutting tool performance (iii) Toughness (iv) Fatigue resistance (v) Damage Tolerance.

The high strain rate repetitive contacts simulate the severe dynamic loading and impact/erosive wear conditions in auto- and aero-engines and high speed cutting.

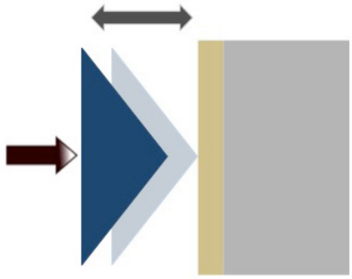

Figure 1. The Nano-impact test involves multiple true high strain rate impacts on the sample surface

Experimental parameters such as probe geometry, acceleration distance, applied load, impact angle, number of cycles and impact frequency can be controlled to alter the severity of the test and its duration. Impact testing is a fatigue process, so repeat tests are necessary to obtain a statistical measure of the coating's resistance to fracture. In contrast to macro-scale impact testing which requires multiple specimens, is very time consuming and not sensitive to surface treatments or coatings testing at the nano-scale enables many instrumented repeat tests to failure to be performed automatically on the same specimen in a very short time. Multiple samples of different composition or preparation or indeed geometry may be simultaneously mounted and tests performed in an automated schedule

Impact resistance of different coatings can be compared by the time required for failure (fracture) to occur in 50% of the tests, enabling comparison between similar materials [3,4]. Fracture probability can be estimated by ranking the time-to-failure events in order of increasing fatigue resistance and then assigning a probability of failure (equation 1)

$$P(f) = n/(N + 1) \quad [1]$$

to the nth ranked failure event in a total sample size of N, to produce a cumulative probability plot, analogously to the treatment of distributions of failure stresses in Weibull statistics. The probability of failure with a given time (usually the end of the test) is a convenient way to compare different samples. By combining fracture probability data at different loads it is possible to produce a graph of the time or number of impacts required for fracture to occur in 50% of tests vs. impact force. The energy supplied for coating failure can be calculated from the time-to-failure data using equations 2 and 3.

Energy per impact = force x accelerating distance [2]

Total energy supplied = number of impacts x energy per impact [3]

For test conditions of 1 impact every 4 seconds the number of impacts to failure = 0.25 x time-to-failure in seconds.

Procedure

Samples

The coating systems studied were (a) compositionally graded a-C:H (H-containing DLC) and a-C (H-free DLC) coatings deposited on M42 tool steel by closed field unbalanced magnetron sputter ion plating (CFUBMSIP) (b) a non-graded a-C:H coating on steel [4-5]. The amorphous carbon coating (Graphit-iC™) had a total coating thickness of 2.5 µm and the hydrogen-containing DLC coating (Dymon-iC™) a total coating thickness of 2.8 µm. For comparison a non-graded a-C:H/Cr coating on tool steel (2.3 µm total thickness) was also tested. All three coatings had a Cr bond layer, of thickness 0.4 µm on the a-C; 1.1 µm on the graded a-C:H and 0.3 µm on the non-graded a-C. The coating architecture for the compositionally graded coatings is shown schematically in figure 2.

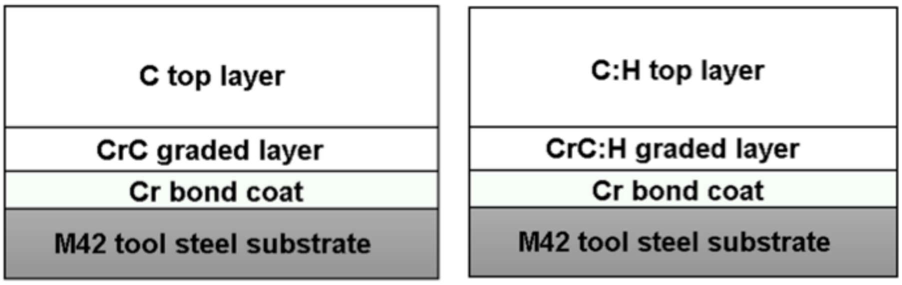

Figure 2. Coating architecture on compositionally graded coatings

Equipment

The modular Micro Materials NanoTest system was used for the nanoindentation and nano-impact testing.

Prior to nano-impact testing the mechanical properties of the coatings were determined by nanoindentation according to the ISO14577 procedure.

Test Method

Prior to nano-impact testing the mechanical properties of the coatings were determined by nanoindentation according to the ISO14577 procedure. Surface roughness was determined by topographic scans (Line Profiles) in the NanoTest scanning module.

Nano-impact tests were performed on graded a-C:H and a-C coatings with a cube corner diamond indenter test probe was accelerated from a distance of 15 µm from the surface to produce each impact at applied loads of 1, 5 or 15 mN. A cube corner diamond indenter whose geometry induces high contact strain is often chosen as the test probe, as this high contact strain is beneficial in inducing fracture within a quite short test time. The experiments were controlled so that repetitive impacts occurred at the same position every 4 seconds. 10 repeat tests, each of 1800 s duration, were performed at different locations on each sample. Impact conditions were identical for the 2.3 µm a-C:H although the test time was shortened to 900 s and the loads chosen were 3, 5, 10 and 15 mN.

Results

Nanoindentation

The mechanical properties of the coatings are summarised in Table 1. The R_a surface roughness of the graded coatings was ~35-40 nm.

Coating	Thickness (µm)	Hardness (GPa)	Elastic modulus (GPa)	*H/E*	H^3/E^2 (GPa)
Graded a-C	2.5	13.9	181	0.077	0.082
Graded a-C:H	2.8	17.0	139	0.122	0.253
Non-graded a-C:H	2.3	21.0	247	0.085	0.152

Table 1 Nanoindentation data for amorphous carbon coatings on M42 tool steel

Nano-impact of graded coatings

The tests revealed clear differences in the coating behaviour under repetitive impact. The graded a-C:H was more susceptible to impact-induced fracture and coating failure than the a-C coating. During the impact tests abrupt depth-steps are observed when coatings fracture. These occurred after only a few impacts on the graded a-C:H coating. This can be more clearly seen by investigating the beginning of the impact test. Figure 3 shows depth vs. time data for the first 240 s of the test, corresponding to the first 60 impacts. Within this time period the a-C coating did not fracture but the a-C:H coating fractured almost immediately resulting in material removal and greater impact depth. With continued impacts the a-C coating did fracture (figure 4) but these were less extensive than on the a-C:H. Figure 4 displays the number of impacts required to cause fracture in the nano-impact tests at 5 mN as a cumulative probability plot. Figure 5 shows the load dependence of the final impact depth for both coatings. The micrographs confirm that fracture is more extensive at higher load and on the a-C:H.

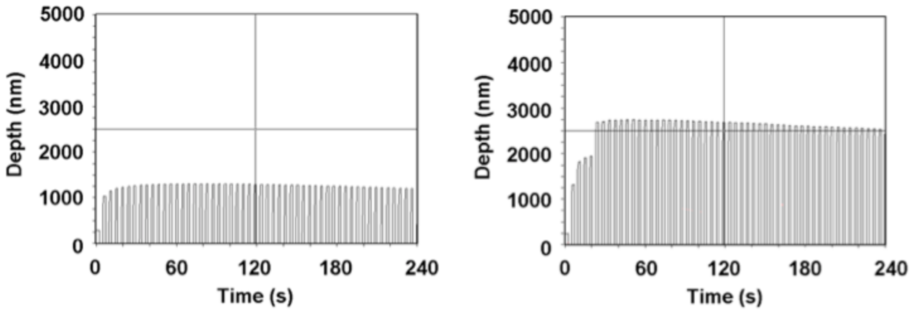

Figure 3. Comparison of impact behaviour at 5 mN (L = a-C; R = a-C:H)

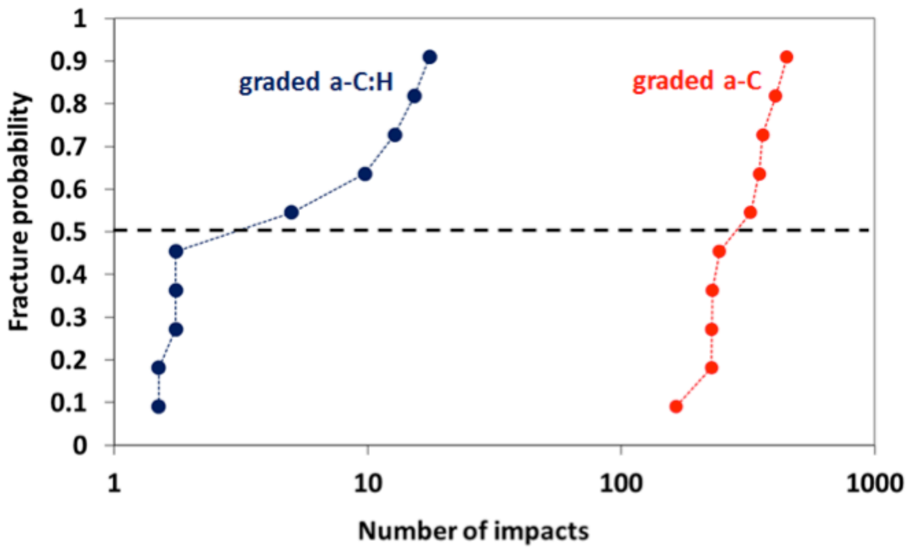

Figure 4. Cumulative probability distribution of time-to-failure in 5 mN tests on graded coatings

The micrographs confirm that fracture is more extensive at higher load and on the a-C:H.

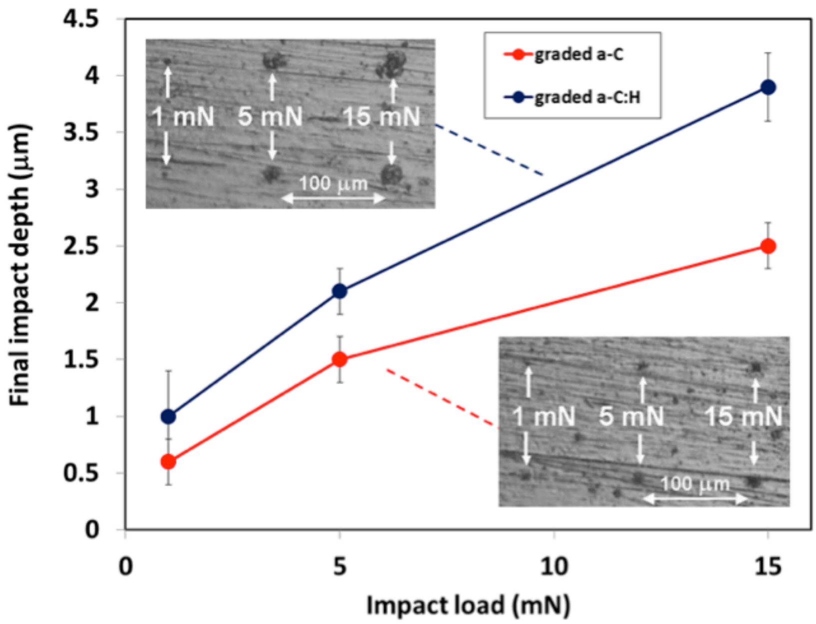

Figure 5. Final impact depth after 450 impacts at 1,5 and 15 mN on graded a-C:H and a-C

Nano-impact of non-graded a-C:H

Figure 6 shows how the applied load influences the time-to-fracture for the non-graded coating. When the impact load is low the coating is durable and when fracture occurs it is cohesive, but above a threshold load the through-thickness cracking occurs readily and leads to removal of the coating (final depth > coating thickness). Coating failure is more rapid and occurs over a smaller number of impacts at higher load. At 5 mN the smaller (cohesive) failure proceeds more gradually.

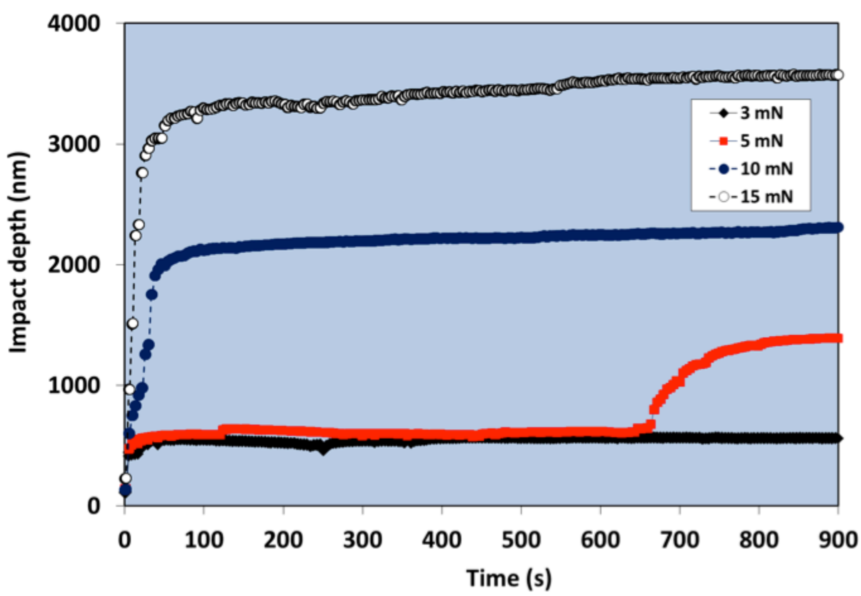

Figure 6. Load dependence of impact wear on a-C:H on tool steel

The a-C coating showed significantly improved impact resistance compared to the a-C:H coatings, despite its lower hardness.

Discussion

The a-C coating is lower in hardness but stiffer and consequently has lower *H/E* and H^3/E^2 than the a-C:H coatings. The higher hardness and sp^3 bonding in a-C:H result in hard, elastic contact and reduced durability while the a-C coating system exhibits enhanced impact resistance. This is consistent with other studies showing an improvement in wear resistance of a-C coatings developed using the CFUBMS process [6], although the nano-impact testing shows the difference more dramatically. Amorphous carbon coatings produced by the CFUBMSIP process are reported to have a graphitic structure, and this sp^2 bonding can impart low stress and improve adhesion. Improved wear resistance has been reported for coatings with high *H/E,* since this is related to the elastic strain-to-break, but under highly loaded conditions there appears to be a benefit in designing coatings with compositional grading, for efficient stress transfer to substrate and some plasticity for stress relief.

Conclusions

The a-C coating showed significantly improved impact resistance compared to the a-C:H coatings, despite its lower hardness. In automotive engine applications involving a combination of load support and resistance to fatigue optimum lifetime of coated components may be achieved by designing the coating system to combine these properties rather than by solely aiming to maximise coating hardness as this may be accompanied by brittle fracture and higher wear [7].

The advantages of the nano-impact test are:

(1) short duration of the experiments compared to conventional tests allowing rapid screening to evaluate the performance of novel coating compositions

(2) multiple rapid tests possible on single samples

(3) flexibility to alter loading level and severity of impact loading

(4) can be combined with the nanoindentation and nanoscratch test modules in the NanoTest Vantage to deliver a complete multifunctional nanomechanical characterisation capability in a single test platform

(5) the possibility to schedule experiments on multiple samples for rapid performance comparison

References

1. R. Gåhlin, M. Larsson, P. Hedenqvist, ME-C:H coatings in motor vehicles, Wear 249 (2001) 302-309.

2. J. Jiang and R.D. Arnell, On the running-in behaviour of diamond-like carbon coatings under the ball-on-disk contact geometry, wear 217 (1998) 190-199.

3. B.D. Beake, S.R. Goodes, J.F. Smith, G.S. Fox-Rabinovich and S.C. Veldhuis, Using nanomechanics to optimise coatings for cutting tools, in Handbook of Nanostructured Thin Films and Coatings, Mechanical Properties, Chapter 6, pp205-244, Ed. S Zhang, CRC Press (2010).

4. B.D. Beake and J.F. Smith, Nano-impact testing - an effective tool for assessing the resistance of advanced wear-resistant coatings to fatigue failure and delamination, Surf Coat Technol 188-189C (2004) 594.

5. B.D. Beake, Evaluation of the fracture resistance of DLC coatings on tool steel under dynamic loading, Surf Coat Technol 198 (2005) 90.

6. X.T. Zeng, S. Zhang, X.Z. Ding, D.G. Teer, Comparison of three types of carbon composite coatings with exceptional load-bearing capacity and high wear resistance, Thin Solid Films 420-421 (2002) 366-370.

7. Nanomechanical characterization of carbon films, B.D. Beake and T.W. Liskiewicz, book chapter in Applied Nanoindentation in Advanced Materials, 978-1-119-08449-5, PP 19-68, Editor Atul Tiwari. Wiley October 2017.

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