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A novel characterization method for hard coatings: preliminary results with TiN

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Abstract. Pin-on-disc method is increasingly used to evaluate the tribological behavior of thin hard coatings. At present, ISO 18535 is the only standard applying to hard coatings tribology and it prescribes that investigations do not extend past the coating failure. This approach followed by many researchers is inadequate though for a complete characterization of a coated system. A novel approach is proposed in this paper, at a preliminary stage, to characterize coated systems thoroughly. Friction curves with a suitable shape are resorted to, so that two representative friction coefficients can be extracted, one before and the other after coating failure. Results with a PVD TiN coating are presented showing that it is possible with this coating to get friction curves with the desired shape in specific testing conditions.

Keywords: pin-on-disc, coatings, characterization method, TiN

1 Introduction

Many modern industrial users are resorting to pin-on-disc (PoD) method to evaluate the behavior of thin hard coatings [1]. In current state-of-the-art, ISO 18535:2016 [3] is the sole standard dealing with PoD tests of thin hard coatings. It was introduced for DLC but it extends to any kind of hard coating. This standard, though, focuses on the tribological behavior of the superficial layer only, and recommends that investigations do not extend past the coating failure. In practice, this approach successfully characterizes films but fails when the characterization of the tribological system is required as a whole. Many researchers follow this attitude, aware or not of this standard, and investigations usually stop before or at coating failure. For some applications it is interesting to know how the coefficient of friction (CoF) evolves after the coating failure, for example to be aware of coating failure without direct surface inspection. Moreover, in some cases it is necessary that mechanical components with protective layers on top could operate after the coating failure over a period, e.g for safety reasons in aircrafts.

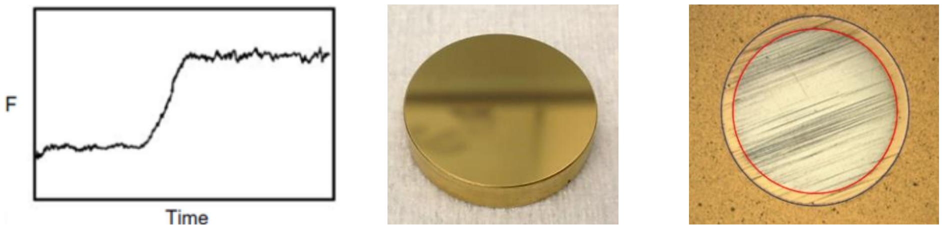
Such a scenario represents the starting point for a new approach in the tribological study of coatings. If stable and sufficiently repeatable friction curves can be obtained with a suitable shape, then a unified method can be proposed to wisely characterize systems featuring coatings. Friction curves must have a suitable shape as the one sketched out in Fig. 1. They should meet three crucial requirements ideally: 1) featuring an obvious transition from coating to substrate contact; 2) disclosing a stable coefficient of friction (stable mean value) before and after the coating failure; 3) ensuring a satisfactory repeatability of the overall shape and the local CoF.

ASTM G99 is the other commonly used standard for PoD tests. It provides a procedural guideline [2] i.e. the modus operandi to carry out tests and calculations. According to ASTM G99, the average, maximum and minimum CoF are enough to describe whatsoever of friction curves. This is useless instead in a scenario like above: two average coefficients of friction, one before and the other after coating failure, are more convenient to characterize the system completely. Doing so prevents friction curves to be addressed as-is and an identification process becomes mandatory to extract the two average coefficients of friction from relevant portions of friction curves.

In this paper, such a novel method was tested in order to make a preliminary evaluation of its feasibility and significance. An extended experimental investigation involving only one hard coating was carried out. A TiN PVD coating was chosen being a ceramic compound largely studied [4] and used for many industrial applications, e.g. to reduce wear of cutting tools and bushings. A basically empirical approach was followed and little insight into the tribological behavior of this specific coating is provided as it goes beyond the scope of this investigation.

Many other research works dealt with tribology of TiN coatings, for example Kara et al. [5], Bienk et al. [6], Yan et al. [7], Monteiro Baptista [8] and Vancoille et al. [9]. The common approach is very different from the one adopted here, anyway. It is not uncommon that authors focus on either friction or wear behavior separately (running dedicated tests) and even if the two are taken into account at once the exploration of friction behavior stops as the coating layer fails.

This paper summarizes a preliminary study in perspective of a broader investigation which will involve a variety of coatings and will have to address a tricky issue that this paper does not explore: what criteria would allow users of this novel method to analyze data and extract the representative CoF values with objectivity?



(a) (b) (c)

Fig. 1. (a) Curve with suitable shape; (b) TiN-coated sample; (c) ball-crater test on a sample.

2 Materials and Methods

Tribological tests were performed by means of an Anton Paar TRB pin-on-disc tribometer in one-way rotating mode. 10 steel discs were machined from a 90MnCrV8 bar and then coated with a micrometric layer of TiN by Physical Vapor Deposition (PVD) technique after rectification and polishing (Fig. 1b). The ceramic coating was produced by Oerlikon Balzer AG. The thickness of the ceramic layer was verified by ball-crater method (see Fig. 1c) with an Anton Paar CAT^{2}c. The average thickness over the 10 samples was found to be 1.77 µm ± 0.09 µm. Coating elastic modulus and hardness was measured with an Anton Paar NHT^3 instrumented nano-indentation tester as well. Values equal to 360GPa and 24,3GPa (equivalent 2251 HV/0.002) were measured on average, respectively. Both nano-indentation and thickness measurements were performed in the central part of samples where tribological tests cannot run. Samples were tested against spheres of different size and material. Sphere hardness was measured by classical hardness test with an Anton Paar RST^3 tester: 1674 HV/1 for alumina balls and 1747 HV/1 for WC balls.

The experimental study was arranged in two phases. During the first phase a broad tribological characterization of the selected coating was performed by testing various configurations, sphere materials and tribological parameters. Several combinations of load ranging from 1N to 6N and linear speed ranging from 0.1m/s and 1m/s were tested. This was to find out an optimum combination of testing parameters, one which provides friction curves featuring an obvious coating- to-substrate transition, a stable coefficient of friction before and after transition and uniform coating wearing-out as well. In the second phase, repeatability of the results obtained with the identified prospective optimum parameters was investigated and a new sample was used for every repetition to keep constant the radius of the wear track. Four repetitions were carried out. Table 1 provides an overview of the optimum parameters identified. It is important for users that tests are not overly time-consuming. This issue was also taken into account to accept the optimum parameters identified.

A great deal of attention was paid to avoid any kind of contamination of the samples surface. The entire equipment was periodically cleaned; the sample and the ball were cleaned by immersion into isopropyl alcohol, dried and wiped with lint-free tissues before starting each test. Temperature and humidity were monitored during experiments with oscillation from 25 to 30°C and 30 to 45% moisture into the testing chamber.

Tab. 1. Optimum testing parameters identified as output of the first experimental phase

Sphere	*Load [N]*	*Speed [m/s]*	*Duration [m]*	*Track radius [mm]*
10mm Al_2O_3	1	0.1	Variable	4.2

3 Results

The first part of the investigation covered more than 70 tests, that allowed to identify the maximum suitable load and speed to be used for the second part of the research activity. Load should allow enough duration of the coating, avoiding early failure at the beginning of the test and non-uniform wearing out. With loads higher than 1N the friction curves disclose all an early perforation of the coating immediately after the wear-in ramp (Fig. 2a and 2c) and the transition phase from coating to substrate run is generally not obvious. In most of the tests, the failure of the coating is strongly non-uniform: traces width changes deeply and areas where coating is totally worn out coexist with areas where coating is still there along the same trace, e.g. Fig. 2b.

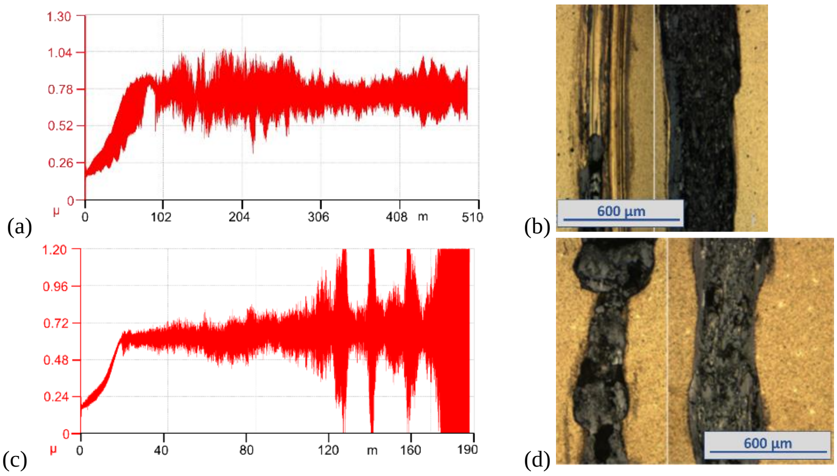


Fig. 2. (a) 5N, r=11.4mm, 0.48m/s, 6mm WC ball; (c) 3N, r=5.3mm, 0.5m/s, 6mm WC ball; (b) and (d) are the corresponding wear traces.

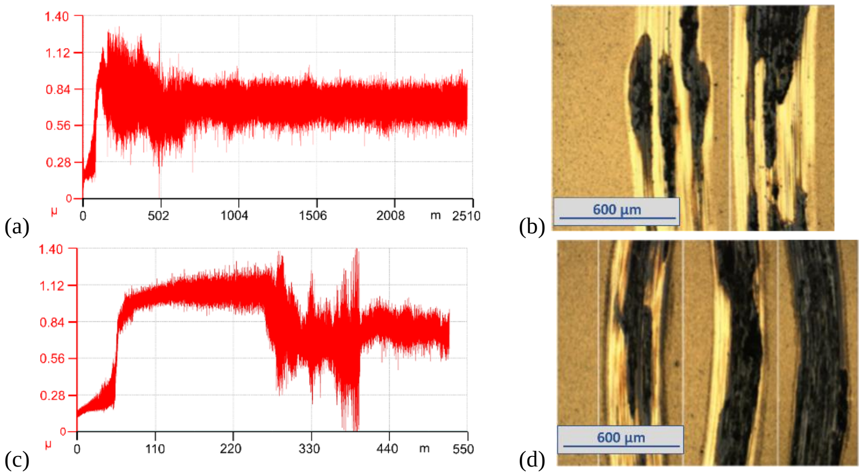


Fig. 3. (a) 1m/s, 1N, r=11.2mm, 10mm Al2O3 ball; (c) 0.5m/s, 1N, r=5.2mm, 10mm WC ball; (b) and (d) are the corresponding wear traces.

Furthermore, high load (especially in combination with high speed) undermines the stability of the measuring arm such that hopping of the pin is common. When coating failure starts this effect is further amplified as the contact path becomes irregular.

TiN is a brittle material and the reiteration of impacts on its surface is likely to induces the nucleation and propagation of cracks (in surface and beneath) until a large debris is ejected: this may explain the occurrence of craters and bumps (Fig. 2d) that are sources of high noise and banging. As soon as these circumstances are met, the test is invalidated because dynamics begins to strongly influence the tribological result.

Fig. 3a-d show tests performed under a constant load of 1N. Similar remarks as those for load apply here for the impact of speed on system dynamics. Strong non-uniformity of the wear scar and irregular perforation of the coating layer cannot be avoided if speed is faster than 0.1m/s. Fig. 3c may represent an acceptable result in term of friction curve, however the transition phase from coating to substrate had not been completed yet (see microscopic observation of traces in Fig. 3d) despite the second CoF value stabilization. This condition was also proven to be non-repeatable as later attempts were carried out.

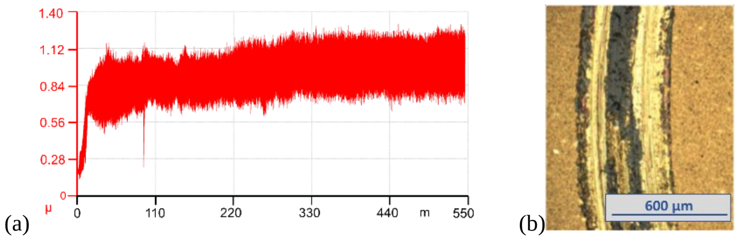


Fig. 4. 6mm WC ball, 0.1m/s, 1N, r= 4.75mm radius test. (a) Friction curve; (b) wear trace.

The only parameter left free is the material of the sphere. Fig. 4 shows a test running with the previously fixed speed and load and a 6mm WC ball. Inspection of the wear trace pointed out that WC was not able to entirely remove the hard layer, at least within a reasonable duration of the tests. Besides, there was evidence of a grey transfer layer that would probably grow as the interaction would go forward.

The analysis of the above preliminary results allowed to identify the optimum parameters for the second part of this study (see Table 1), where the same test was repeated four times (each time on a new sample) to verify its repeatability.

Tab. 2. Output of the four repetitions with optimum parameters

	Thickness [um]	*Average COF*	*Maximum COF*	*Minimum COF*	*Duration [m]*
Test 1	1.85	0.948	0.948	0.212	450
Test 2	1.69	0.904	0.904	0.156	530
Test 3	1.89	0.937	0.937	0.220	465
Test 4	1.73	0.906	0.906	0.211	375

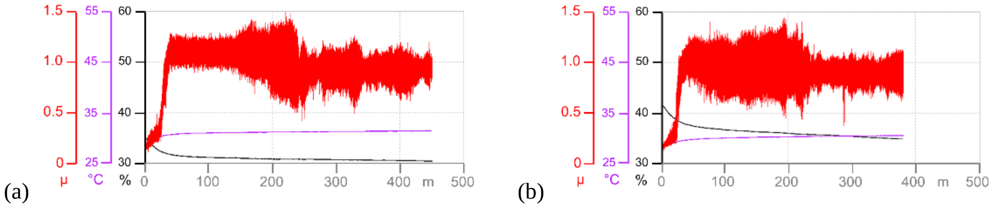


Fig. 5. Example of 2 friction curves out of 4 repetitions of the tests with optimum parameters.

Table 2 summarizes the results of the four repetitions; results are still presented according to ASTM G99 guidelines. For short, Fig. 5a-b show the friction curves corresponding of two out of 4 tests. Tests were stopped after transition being completed so that the substrate wearing out lasted as long as the coating wearing out. All the tests exhibited a fairly good repeatability in fact and similar features emerge at a glance. No measurable wear exists for the ball, just some oxidized debris coming from the sample substrate and welded on it are visible under the microscope. A black region appeared in the middle of the wear trace as well; stylus profilometry was also performed confirming that this black region corresponds to locations where the ball reached the steel substrate.

4 Discussion

It is clear however that table 2 brings no relevant information. The representation of the output as prescribed by the actual release of ASTM G99 is totally inadequate for coated systems which are likely to have a friction curve with complex shape. Further analysis of friction curves is needed in order to obtain the two representative CoF values.

Raw curves in Fig. 5 are quite noisy which makes it difficult to see the transition from coating to substrate. The main stages of the wear process can be perceived but the precise identification of their start and end point is impracticable. Curve filtering may apply, therefore.

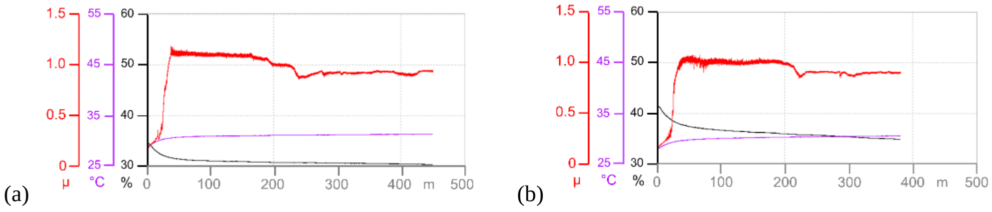


Fig. 6. Filtered friction curves corresponding to raw friction curves in Fig.5

Fig 6a-b presents the curves corresponding to those in fig 5a-b where a 20-points moving averaging filter was applied. Fig 7 sketches out the wear process stages and provides a representation of corresponding relevant portions of the friction curve:

I. A wear-in phase takes up the first 50m of test. The coefficient of friction evolves reaching 1-1.1 from an initial value of about 0.2. At the very beginning, the test run smoothly and the value of the coefficient of friction increases slowly. The trend changes then after 10 to 30 meters and the value peaks with a steep ramp; white debris (TiO2 [5]) starts also appearing around the wear track. The predominate tribological mechanism is probably changing and severe abrasion takes over as the strong increase in CoF suggests [10].

II. A first steady-state period corresponding to the on-coating run. The CoF mean value is stable but scattering may be higher than elsewhere. During the first steady state (*on-coating run* in Fig.7), the uniform wearing out of the ceramic coating takes place. Its duration is variable as it correlates to the unavoidable variability from sample to sample of the ceramic layer properties, thickness firstly. The interaction between the ball and the surface is still smooth and quiet, just a low-intensity whistle may arise. Stages I and II merge in the so-called *coating lifetime*.

III. A *transition phase* of variable length which lessens the coefficient of friction towards a new stable mean value. The behavior of the system is often characterized by a transitory increase of the curve noise, i.e. a larger scattering of CoF. This stage is inhomogeneous; its duration varies from sample to sample, as well as the tribological behavior. Black debris appears on the sample surface (Fe-O and Fe-Ti-O compounds, maybe [5]), proof that steel substrate wearing out has started.

IV. A second steady-state period (*on-substrate run* in Fig.7). It persists until the end of the test. The curve is, on average, thinner besides unregular (raw data). The trend is nearly horizontal, and a new quiet running condition settles in all the repetitions. The sphere is here partly in contact with the substrate and partly with the ceramic film. The presence of a black area in the middle of the track means that oxidation effects took place.

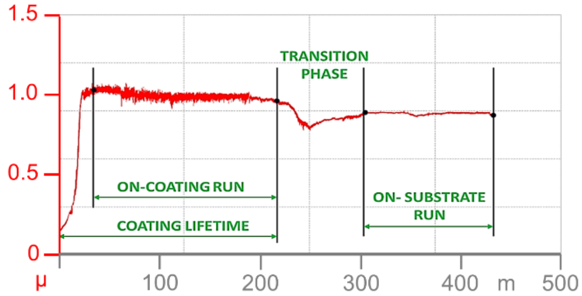


Fig. 7. Wear process stages with representation of the relevant portions of the friction curve

Contrary to what was expected, the mean friction value is higher in the first part of the test (when the ball is in contact with TiN) and decreases in the second part. TiN is primarily an anti-wear coating and usually exhibits low friction when sliding against itself [4]. The function of a hard coating is to protect the substrate and prevent strong ploughing, thus reducing wear. On the other hand, the hard layer has higher shear strength which may increase sliding friction if no microfilms are formed. This can explain why hard coatings can exhibits high friction coefficients under dry sliding [4].

5 Conclusions

Heterogeneous behaviors and phenomena play a role when testing coated components. If the concern is not limited to study the behavior of the ceramic film but to characterize the coated system in its entirety, a new specific approach different from both the ASTM G99 standard and the ISO 18535 standard is necessary.

The output of this work suggests that this novel method is applicable for this coating:

- Optimal testing parameters can be set out by trial-and-error procedure in order to get repeatable friction curves with suitable shape.
- Curves with suitable shape show two nearly horizontal portions of the friction curves where CoF is stable and an obvious transition from coating to substrate in between.
- The nearly horizontal portions of the friction curves are the relevant portions which provide the two mean CoF values characterizing the tribological behavior of the system; that evaluation of the coating lifetime is possible too.

- Filtered curves must be resorted to for the sake of the precise identification of the two relevant portions.

This work is however a preliminary investigation which lays out the foundations of this novel method only. Its complete formalization requires the definition of a robust and unequivocal identification procedure of friction curves in the future. This conventional procedure may be based on a graphical technique and might also involve other mechanical properties of the coating. Without this, the whole analysis of friction curves would be prone to inconsistency and there would be the risk for it to come down to the user subjective feeling or understanding.

Subjectivity of the analysis must be kept as small as possible, nevertheless all the quantities that would result from friction curves analysis (CoF before and after coating failure, coating lifetime, ecc…) would be conventional values constituting a basis for the relative evaluation of different kinds of coatings among one another. This is consistent with what should be the correct use of pin-on-disc method [11].

The optimum parameters presented in the previous section are suitable for the specific coating chosen for this investigation. There is no evidence that the same parameters would work with other kinds of coatings or with different coating thickness. Therefore, the authors are already planning a further preliminary study to attempt applying the same method to other coating compounds.

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