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Tribological analysis of thin films by pin-on-disc: Evaluation of friction and wear measurement uncertainty

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ABSTRACT

Pin-on-disc is widely used to evaluate tribological properties of thin films. However, the results are often present without standard uncertainties; moreover, in many cases the standard uncertainty is replaced by standard deviation, which is a strong underestimation of real uncertainty. In this study we have followed ISO and NIST guidelines to investigate the possible sources of uncertainties related to friction and wear rate measurement and to apply them on two selected coating systems – TiN and DLC. We show that influence of operator is a significant contribution to the uncertainty of the wear rate, particularly in the case of very low wear of DLC coatings. We discuss why variance should be used instead statistic deviation and suggest a method to calculate uncertainties in case of small number of measurements. The paper could be used as a guide to evaluate friction and wear data of thin films and coatings using the pin-on-disc technique.

1. Introduction

The experimental evaluation of friction coefficient and wear rate using pin-on-disc is a common laboratory procedure. Despite the simplicity of measurement and calculation, there are practical challenges to quantify these basic tribological parameters accurately. Friction is a typical non-equilibrium process and sliding often leads to wear, which is highly stochastic. The values of friction coefficients and wear rates reported in the literature typically show wide variation even for nominally identical tests; the origin of these variations is often not known. To assess uncertainty of tribological measurement is thus a complex problem. Due to the high spread of measured data, a high number of identical measurements is required to estimate values of friction and wear. The tribological measurement is a lengthy and expensive process; therefore, an optimum number of repetitive measurements must be found to satisfy both precision and economy of the testing. Moreover, in some cases the number of samples and thus number of available tests is limited.

Tribological analysis of thin protective films is in many ways different from that of bulk materials. The thickness of the film is in the range 0.1–10 μm with 1–3 μm being the typical value. The films are quite often composed of bonding interlayer improving adhesion (metals, carbides, nitrides, gradient interlayers) and top functional coating. To evaluate the latter the maximum wear depth is limited to approx. 80% of its thickness to avoid influence of bonding layer. As a consequence, the worn volume is very low and traditional measure of material mass loss cannot be used; thus, mechanical and optical profilometry is required. In some cases the wear is extremely low and the depth of the wear track is close to surface roughness, which leads to high uncertainty of the wear rate.

Unfortunately, the standard procedures [1,2], which should be used to estimate measurement uncertainties, are not always followed. As a consequence, the friction and the wear rate values are often presented without measurement uncertainty; moreover, the uncertainty is sometimes replaced by standard deviation, which is misleading and significantly lower than standard uncertainty.

Uncertainty of tribological measurements has been addressed in several papers for various measurement conditions [3,4,5]. Detailed uncertainty analysis of low friction coefficient measurements with a reciprocating pin-on-disk tribometer has been shown in Refs. [6,7]. In these studies the predominant source of variations originated from the misalignment of the force transducer axis relative to the specimen surface. Nevertheless, the scatter of friction coefficient values was larger than estimated uncertainties related to the experimental apparatus. Krick et al. [8] examined the influence of the ratio of the wear track radius, r, and contact width, $2a$, on uncertainty of friction coefficient measured

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by pin-on-disc. They concluded that the increase of uncertainty was significant only for very small wear track radii. For $r/a \geq 4$, the relative uncertainty was lower than 1%.

In this paper we follow guidelines provided in Refs. [1,2] to analyze in detail the uncertainty of friction coefficient measured by the standard pin-on-disc apparatus and the corresponding coating wear rate. Then we report application of the method to two large set of substrates, one coated by titanium nitride (TiN), the second with hydrogenated diamond like carbon coating (DLC). We determine the most significant contributors to the overall measurement uncertainty, which could help to either re-design the experiment procedure to reduce the measurement uncertainty or to simplify it by neglecting some parameters. We show that estimation of uncertainties could help to distinguish between random value variation and true trends (i.e. dependence of measured values on selected variable or set of variables). Finally, we suggest an optimum process to estimate uncertainties.

2. Measurement uncertainties

The standard uncertainty of measurements is determined using Type A and Type B uncertainty evaluations [1,2]. To evaluate Type A uncertainty the measurement is repeated under the same conditions and the statistical methods are applied to the set of measured values. However, the tribological tests are destructive and the test cannot be repeated under the repeatability conditions stated in Refs. [1,2]. It is clearly demonstrated by the wear rate data dispersion for which orders-of-magnitude variations are common [9]. Thus, the results of the set of measurements cannot be (at least in general) treated with statistical methods; in other words, uncertainties Type A cannot be evaluated. Nevertheless, the testing procedure involves some steps, such as instrument calibration, which fulfill the repeatability conditions and therefore could be evaluated by means of a statistical methods and Type A uncertainty could be determined. The standard uncertainty of tribological measurement is dominated by Type B uncertainties. The uncertainty Type B is evaluated by an engineering and/or scientific judgment based on all available information. In our case it is the estimation of instrument and method errors and operator induced uncertainties.

2.1. Standard uncertainty of the friction coefficient

In this study we consider traditional pin-on-disc tribometer with a ball pressed against a rotating sample (Fig. 1(a)). The pin 1 is mounted on a stiff lever 2, designed as a frictionless force transducer. The dead weight 3 produces the normal force F_n. The friction force F_f is evaluated from the deflection of the elastic arm 4 measured by inductive displacement transducers 5; the calibration referred to above is used to calculate force from measured deflection.

If $u_{\mu A}$ and $u_{\mu B}$ denote the Type A and Type B uncertainties, the standard uncertainty u_μ of the friction coefficient μ is given by [1,2]

$$u_\mu^2 = u_{\mu A}^2 + u_{\mu B}^2. \quad (1)$$

Since the friction measurement cannot be repeated under identical conditions due to progressive destruction of the surfaces in the contact, Type A uncertainty is related only to the calibration procedure. Calibration is provided by a dead weight (5 N) applied to a ball holder (Fig. 1(b)) giving offset for frictional force gauge (zero load is obviously used as the second point). However, it should be pointed out that the calibration could be only considered as an uncertainty Type A provided it is carried out before any individual measurement. In normal testing practice it is not the case – the equipment is calibrated after a certain number of tests

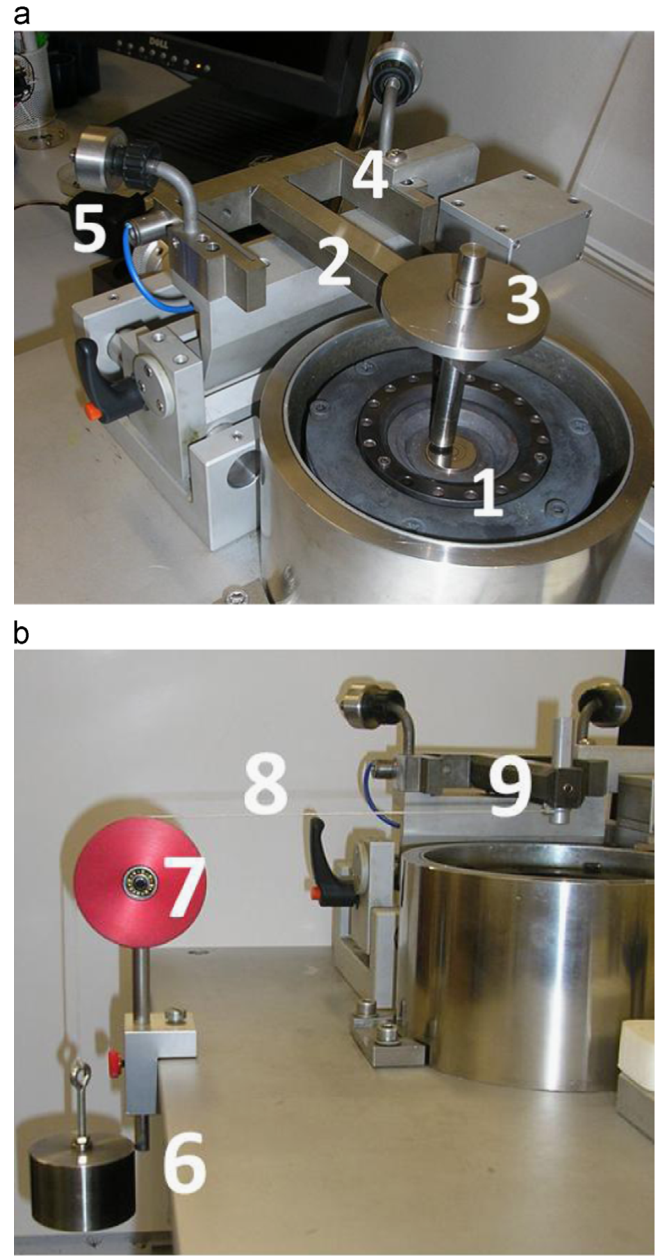

Fig. 1. Tribometer measuring head and scheme of calibration: 1 – pin, 2 – stiff lever, 3 – dead weight, 4 – elastic arm, 5 – inductive displacemant transducer, 6 – dead weight 5 N, 7 – pulley, 8 – string, 9 – pin holder (stiff lever).

or when the material couple is changed. This practice is reasonable when the friction offset (and thus uncertainty Type A) is much lower than total uncertainty of friction coefficient. We carried out number of calibrations giving statistical set of frictional force offsets; standard deviation of the data was then used to estimate uncertainty Type A denoted $u_{\mu A}$.

Based on our experience in the field of tribological measurements we assume the uncertainty Type B consists of instrument uncertainty and uncertainty given by the dispersion of measured values. The origin of the latter is not known; however, it can be estimated on the basis of data difference. We can thus summarize that the Type B uncertainty $u_{\mu B}$ is given by

$$u_{\mu B}^2 = u_{\mu\ i}^2 + u_{\mu\ v}^2, \quad (2)$$

where $u_{\mu i}$ is the instrument uncertainty and $u_{\mu v}$ is the uncertainty due to data difference.