Wear 328-329 (2015) 77–88

Contents lists available at ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

In situ running-in wear assessment in multi-asperity nanotribology

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ARTICLE INFO

Article history:
Received 16 September 2014
Received in revised form
15 January 2015
Accepted 19 January 2015
Available online 27 January 2015

Keywords:
Running-in
Multi-asperity nanotribology
Nanowear
Tribolayers
Nanoindentation
Thin coatings

ABSTRACT

Wear analysis at the micro/nanoscale appears as a great challenge for MEMS/NEMS devices. At this scale classical *post mortem* analysis – like profilometry or AFM assessment – often failed because (i) the error in wear assessment owing to the elastic recovery is no longer negligible at this scale and (ii) the presence of nanometric *tribolayer* within the contact cannot be take into account when the contact is opened afterward. So, this paper deals with an *in situ* wear assessment based on a *triboscopic* approach where the final position z_f of the ball is known without opening the contact because its vertical position is assessed at every instant of the process. This *triboscopic* assessment considers the initial approach of the surfaces z_0 and gives the wear rate by taking into account the presence of any *tribolayer* within the contact. It requires some corrections as (i) the tilt of the sample and (ii) the initial displacement of the surfaces, which is a function of the mechanical properties of the samples. The latter are determined by using an inverse method combining spherical nanoindentation and boundary element numerical simulations, which are both described too. Validation and application of this *in situ* approach to the running-in wear assessment of thin *soft* and *hard* coatings currently used in MEMS manufacturing are finally presented.

1. Introduction

At the micro/nanoscale wear analysis is a great challenge, especially for MEMS/NEMS devices that can be strongly affected by various surface phenomena, such as friction/stiction, micro- and nanoscopic wear, surface contamination and environmental effects [1–8]. Besides, nanowear mechanisms met in various microsystems are generally closer to the *polishing* process in terms of wear rate [9] than the classical mechanisms of *abrasion* [10], *adhesion* [7] or *ploughing* [4,11,12]. This peculiar wear process mainly results from the combination of a low contact pressure and a *closed* multi-asperity tribocontact that acts as a *triboreactor* at the micro/nanoscale [13–17], where thermal effects [18], chemical and physico-chemical interactions [19,1,20,21,7], environment's influence [14,22–24,15,19,25], and *tribolayer* are likely to control the tribological behavior, as a self-organization process [26,27,15,16,18,17].

Currently, topographical assessment is commonly used for analyzing wear at this scale [3,2,11,28,29]. Wear is then measured by determining the depth (or volume) of the wear track at the end of the test with a profilometer [10,30,21,23,31,15,16]. However, it is worth noting that this *post mortem* assessment is only suitable when the wear level is much more significant than the deformation one. Otherwise the error on the wear value due to the elastic recovery is no longer negligible [30,32,31]. In addition, this approach clearly assumes that only the *mechanical* component of wear occurs. This is generally true in a LFM/FFM experiment that simulates a *mono-asperity* contact with an AFM tip [10,21,33,34,17,35], but not any more in a real MEMS displaying many asperities [1,20,36,37,7,22,38,25,17]. In the latter case, *physico-chemical* aspects are superimposed on the *mechanical* ones. As a result, *adhesive* wear component also occurs leading to the formation of a self-organized *tribolayer* that usually controls both the friction and wear behaviors [39,27,26,15,16,18]. Thus, whatever its intrinsic accuracy [32,30], the main drawback of the topographical assessment is that any measurement is always carried out after the contact opening, meaning the loss of all the existing relationships between the frictional evolutions and the wear events. Hence, while this classical *post mortem* approach generally fails in the presence of such *tribolayer,* a real-time wear assessment would be likely to consider any *dynamic* influence of *tribolayers* on the tribological behavior [40,14,23,24,17].

Interesting ways to study a continuous running-in tribological system in real-time were recently proposed by Scherge et al. [41,9] and Dienweibel et al. [42]. The former describes an *in situ* wear assessment that is based on a radionuclide-technique, as a thin layer activation method [41] while the latter, rather proposes a real-time wear assessment using an on-line topographical approach [43]. All these approaches might work successfully in the case of

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http://dx.doi.org/10.1016/j.wear.2015.01.033

MEMS because of their own accuracy and extremely high resolution (typically 0.1 nm/h).

As a simpler alternative, this paper describes an *in situ* approach which allows us to study wear mechanisms occurring on the micro/nanoscale level by considering a *multi-asperity* contact under low contact pressures (several hundreds MPa) and relatively high velocities (several mm/s) as met in MEMS [17]. This *triboscopic* approach considers the initial deformation of surfaces and gives the wear rate by taking into account the presence of any *tribolayer* within the contact. This *in situ triboscopic* approach will be detailed in Section 2 and validated on reference samples (pure silver and gold) in Section 4. The method will be finally apply to the case of thin *soft* and *hard* coatings currently met in MEMS manufacturing [44–46].

2. Theoretical background and practical approach

2.1. Triboscopic approach

Let us consider an experimental device [17] consisting of a *ball-on-disc* nanotribometer in a linear reciprocating mode (see Fig. 1 and details in Section 3.1), which simulates a typical MEMS tribocontact – as a *comb drive* for instance [1,20,5]. A Si_3N_4 ball (Ø 1.5 mm) is mounted on a stiff lever designed as a frictionless force transducer and loaded onto a flat sample with a precisely known force using a closed loop. The friction force is determined during the test by measuring the deflection of the elastic arm. A real-time depth measuring optical sensor is used for studying any vertical displacement of the ball along the friction track (Fig. 1).

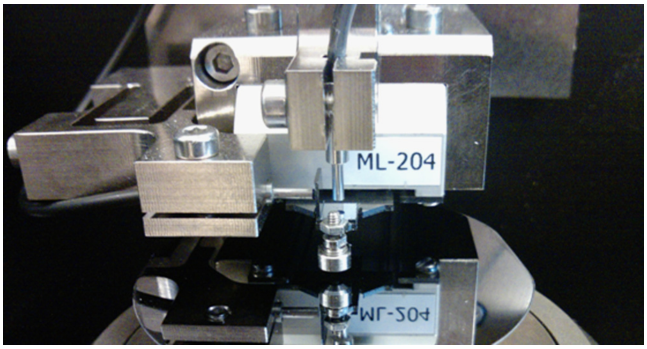

Fig. 1. Ball-on-disc nanotribometer and its real-time depth measuring optical sensor.

Results can be then compiled as a *triboscopic* approach giving simultaneously, and for each cycle:

- the *friction map* (Fig. 2a) plotting the evolution of the friction coefficient along the friction track;
- the so-called *depth map* (Fig. 2b) revealing any time-dependent wear process and/or potential build-up of a *tribolayer* within the contact [17]. However, this *depth map* cannot directly be assimilated to a real *wear map* because it includes: (i) the titling of the flat sample and (ii) a wrong value of the initial vertical displacement which actually corresponds to the initial elastic–plastic displacement of the flat sample under the initial applied normal load (so-called z_0).

Thus at the micro/nanoscales, and in contrast to the classical approaches of macroscale wear assessment, a preliminary accurate knowledge of the elastic–plastic behavior of samples is necessary because the deformation process strongly affects the precision of the *wear map* initialization. Keeping this consideration in mind, the nanotribological process – leading to the *depth map* (Fig. 2b) – can then be decomposed into four stages as follow:

- *Stage* 1: The *loading* that involves a vertical displacement z_0 from the initial surface. This value corresponds to the actual *zero-wear* vertical position. It can be computed by means of numerical simulation [47–49] – as a classical indentation problem of spherical punch on flat sample – as soon as the mechanical properties of each of the contact antagonists are known (*i.e.*, ball, coating and substrate in a general case).
- *Stage* 2: Starting from z_0, the *sliding* involves an evolution of the instantaneous vertical z_i as a function of both the lateral displacement and the number of cycles. Thus, any variations of z_i can reveal abrupt changes in the wear process. Note that z_i normally increases when wear process occurs but it can also decrease – and even become lower than the initial displacement z_0 – in the presence of a *tribolayer* within the contact [17]. In practice z_i can be extracted from data provided by the real-time depth measuring optical sensor from the *triboscopic* approach. However, since tribological tests are carried out in a linear reciprocating mode, a post-treatment of the raw depth data is needed in order: (i) to separate data from the *forward* and *backward* signals and (ii) to correct the sample tilt error. By considering the sign of the sliding velocity along the friction track, two *depth maps* (Fig. 3a and b) can be extracted from a *GNU Octave* script (http://www.octave.org/) and studied separately with the topographical software *Gwyddion* (http://gwyddion.net). Using the latter, sample tilt error along the friction

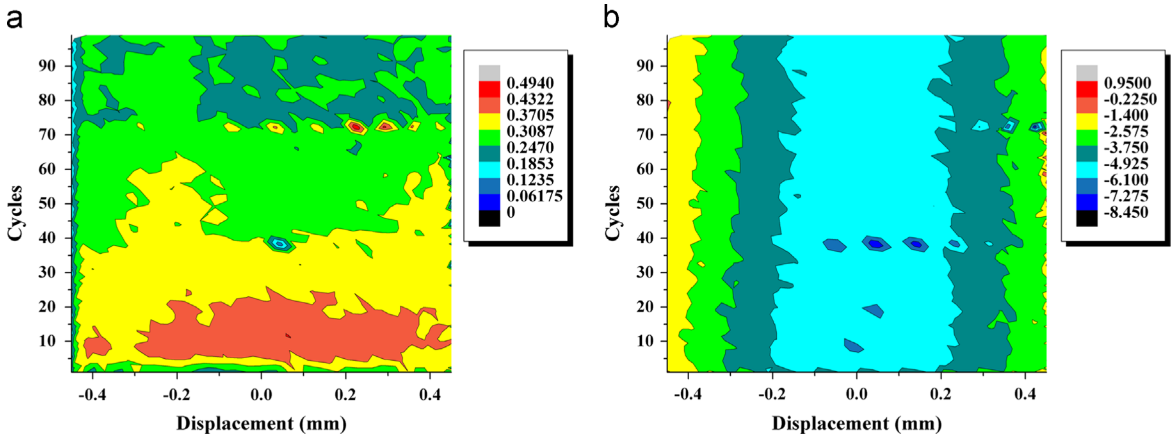

Fig. 2. Typical views resulting from a *triboscopic* approach giving simultaneously and for each cycle: (a) the evolution of the friction coefficient along the friction track (so-called *friction map*) and (b) the evolution of the ball depth within the friction track (the so-called *depth map* in μm). Samples: Si_3N_4/Silver – 45 mN – 100 cycles – 1 mm s^{-1}.