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Mechanical and chemical wear components in environmental multi-asperity nanotribology

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ABSTRACT

Running-in wear of MEMS can be described as a process which involves mechanical, chemical and physico-chemical phenomena at various scale levels. Extracting each sort of components would enable to better understand wear mechanisms in order to prevent it. Unfortunately, these components are generally hard to extract experimentally because their own time responses are generally not in the same order. A suitable approach is to combine multi-asperity nanotribological tests, using an *in situ* wear assessment, with numerical simulations using Movable Cellular Automata (MCA), which are able to interact together within the contact. Experimental tests enable to control the actual physico-chemical environment with an environmental enclosure, and MCA simulate the multi-asperity contact, where interactions between automata pairs can be controlled by various *fracture* and *bonds criteria*. There is generally an *optimal set* of interaction criteria which provides numerical results that match correctly with the experimental ones. By studying the influence of experimental environment on this *optimal set*, assumptions can be made about mechanical, chemical and physico-chemical phenomena that are likely to occur within the actual tribocontact. In this work, these assumptions have been studied for various samples like silicon wafers displaying various crystallographic orientations and nanostructures, and self-assembled monolayers grafted on silicon wafers, and carbon nitride coatings rubbing under different environmental conditions.

1. Introduction

Running-in wear of MEMS involves mechanical, chemical and physico-chemical phenomena at various scale levels – *i.e.*, fracture, ploughing, seizure, adhesion/stiction and also agglomeration of nanodebris as cohesive *tribolayers* [1–5]. Extracting each sort of components would allow to better understand wear process from the macroscale down to the nanoscale level, in order to prevent it [6]. However, chemical and mechanical wear components are generally hard to extract experimentally because their own time responses are generally not in the same order [6–8]. Hence, mixing chemical reactions and mechanical deformations in a same wear model is quite difficult to carry out, whereas these mechanisms work together rather well in the real world. A suitable approach to do this consists to combine nanotribological tests [2–4,9–13] and discrete element [7,8,14–19] or Movable Cellular Automata simulations [20–25]. Indeed, whereas experimental tests enable to control the actual chemical environment by fixing atmosphere, amount of moisture, temperature or partial pressure in a glove box [12,13], numerical simulations give the opportunity to access to the *hidden* parameters controlling *agglomeration* and *fracture* processes, which occur during tribological tests [7,20–24]. Thus, the knowledge of these *hidden* parameters – and especially their variations versus the environmental parameters – seems to be a right way for understanding the various mechanical and chemical components of the involved wear process. However, it is well known that classical mono-asperity nanotribology – using LFM/FFM experiments – usually favours mechanical component of wear [2,4,9,10,26,27] – as *ploughing*, *cutting* or *wedging* – to the detriment of the physico-chemical ones, leading to awear assessment, which is very different to what it is generally observed for typical MEMS tribocontacts [1,3,12,13,28,29].

Thus, in this work, multi-asperity nanotribological tests have been chosen in order to allow any type of wear process including *polishing* and *adhesion* process [13]. Indeed, a closed multi-asperity tribocontact acts as a *triboreactor*, where mutual interactions coming from all neighbouring contacts (so-called *real contact area*) are likely to occur with the environment [30]. Hence, a continuous flow of molecules from the environment can both react chemically with fresh debris created *in situ*, and modify the mechanism of particles detachment itself by means of various mechanical or chemical processes. As a result of combined mechanical and physico-chemical processes, friction and wear behaviours can be analysed from an *in situ* triboscopic approach [31,32].

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In parallel, numerical simulation needs to model this *triboreactor* at the scale of the *real contact area*. Hence, the model has to take into account both the generation of debris, as a wear process involving fracture mechanisms, and their recycling within the contact, as a *tribolayer* generated by an agglomeration process [6,33–35]. Only the discrete element approaches allow to do this because interactions between particles can be controlled by various *fracture* and *bonds formation criteria*, as demonstrated by numerous authors with various methods [7,14,15,18–25,36]. Trying various sets of *interaction criteria*, there is generally an *optimal* set providing numerical tribological results, which well match with the experimental ones. Thus, by studying the influence of experimental environment on this *optimal* set, assumptions could be made about mechanical, chemical and physico-chemical phenomena, which are likely to occur within the actual tribocontact.

In this work, these assumptions will be studied for various samples – bare silicon wafers and self-monolayers grafted on silicon wafers displaying various crystallographic orientations and nanostructures –, which are submitted to different temperature and amount of moisture. Finally, a systematic procedure – providing internal parameters that control *fracture* and *agglomeration* mechanisms during wear process – will be proposed and tested on carbon nitride coatings by using design of experiments (DoE) in order to take into account the experimental uncertainties.

2. Multi-asperity nanotribology

2.1. Experimental set-up

The experimental device is constituted by a *ball-on-disc* nanotribometer manufactured by *CSM Instruments* (Switzerland) [13]. A pin is mounted on a stiff lever, designed as a frictionless force transducer ($K_x = 265.1$ N m^{-1}; $K_z = 152.2$ N m^{-1}). The ball (Si_3N_4 – ⌀1.5 mm) is loaded onto a flat sample with a precisely known force using closed loop. The friction force is determined by measuring the deflection of an elastic arm designed as a frictionless force transducer (resolution: 1 μN). Tribological tests are carried out in linear reciprocating mode under controlled environment using a glove box (Fig. 1a). The relative humidity can vary from 0 to 90% with a saturated aqueous solution of $CaCl_2$ maintained at various temperatures from 3 to 50 °C [12]. Temperature of samples is controlled from −5 to 80 °C by using a Peltier device (cf. Fig. 1b).

2.2. Triboscopic approach

Experimental results are compiled to give simultaneously the variations of both the friction coefficient (cf. Fig. 2a) and the depth *vs.* time by means of a real-time depth measuring sensor displaying a resolution of about 20 nm in a range in-between 20 nm to 100 μm [32]. Depth variations are then converted in real wear variations (Fig. 2b) by taking account of both (i) the initial deformation and (ii) the tilt of samples, as described in a previous paper [32]. Finally *wear profile* (cf. Fig. 2c) can be extracted anywhere along the friction track from the *wear map* (Fig. 2b). As shown in Fig. 2, this *in situ* method reveals any time-dependent wear process and/or potential build-up of a *tribolayer* within the contact as demonstrated in [13]. While a decrease of the *wear profile* corresponds to a wear process, an increase can be associated to any agglomeration process. So, the latter generally leads to a wear rate reduction in agreement with the tribological circuit concept, e.g. [37].

2.3. Samples

Tests were carried out with various samples that are generally used in MEMS manufacturing [1,13]. Sample properties are compiled in Table 1:

- Unpolished and polished *p*-doped single-crystal silicon (100) and (111) wafers grown by CZ process. Note that crystallographic orientation of bare wafers changes both their mechanical [38,39] and their physico-chemical properties [40] in relation with their specific atomic structure;
- *n*-alkyltrichlorosilane monolayers (OTS) grafted on polished (100) and (111) silicon wafers. The detailed grafting procedure was described in [12]. Cleaned substrates are immersed in a 25 mM solution of silane in toluene (99.5%, anhydrous) for 20 min at room temperature (22 °C);
- 3 μm carbon nitride (CN_6) coatings deposited on monocrystal (111) silicon wafers (RMS = 0.355 nm) by the PE-CVD technique. Coating procedure was detailed in Refs. [13,41].

3. Numerical simulation of multi-asperity tribocontacts

3.1. Movable Cellular Automata method (MCA)

The Movable Cellular Automata (MCA) method [36] is based on conventional concept of cellular automata (CA), which treats the medium as an ensemble of interacting objets. The dynamic of automata are defined by their mutual forces while their evolution in time and space are governed by the equations of motion. This approach takes advantage of both classical cellular automata (CA) [43–46] and discrete element methods (DEM) [17,14,15,19,18,47] by including the direct simulation of materials fracture – as damage generation, crack propagation, fragmentation, and mass mixing [36]; processes that are known to be difficult to simulate by means of continuum mechanics methods [47,48]. Indeed, while

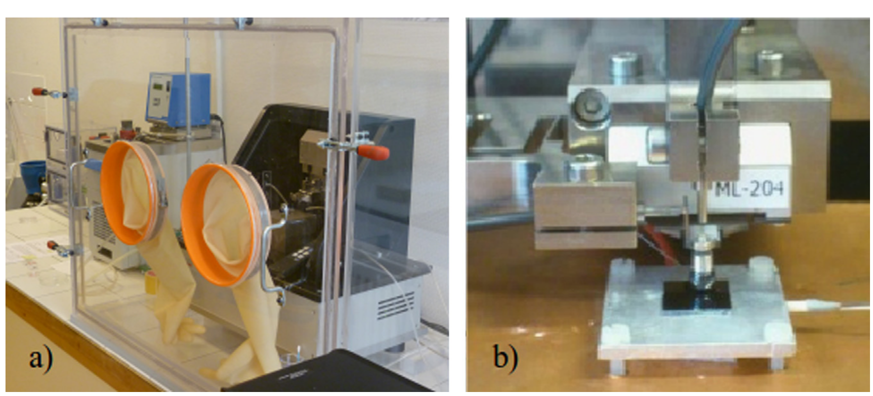

Fig. 1. (a) Environmental nanotribometer in a glove box and, (b) Peltier module.