

# Friction and nanowear of hard coatings in reciprocating sliding at milli-Newton loads

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## Abstract

The wear occurring at very low normal loads and in very small contacts is of prime interest to the field of nanotribology. Friction and wear phenomena in micro-electromechanical components (MEMS) are not well understood and that limits the development of commercial nano components.

In this work, wear and friction at loads in the milli-Newton range was investigated under reciprocating sliding where wear and dissipated energy are in the range of nanometers and microjoules, respectively. Reciprocating sliding tests were performed with a modular micro-tribometer that was operated at normal forces of milli-Newton. This tribometer bridges the gap between macroscale test equipments and the atomic force microscopes. Nanowear tests were carried out for different test durations on hard coatings like DLC and TiN, with silicon nitride balls as the counterbody. After the reciprocating sliding tests at very low loads, the wear tracks were investigated with an atomic force microscope to observe topographical changes in the wear tracks, and to analyze the nanowear. The importance of AFM for characterizing the nanowear appears clearly from this work. The obtained results are compared with existing theories on friction and wear to observe their validity in low load range. The importance of contact pressure and third body interactions in the wear track is also discussed based on AFM observations.

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## 1. Introduction

Conventionally, the simulation of friction and wear at laboratory scale occurs at high normal forces or contact stresses, namely in the order of several Newtons or some GPa contact stress. Pin-on-disk and reciprocating sliding tests [1] are two of the most used tribological test instruments for testing at high normal forces. Friction and wear mechanisms occurring at high normal forces are well established for different materials and coatings, and such highly loaded sliding tests are relevant for instance for cutting tools. The dissipated frictional energy in such tests is very high and gives rise to considerable wear rates in the order of micrometer per hour. As a result, an abundance of third body particles is created, influencing

the contacts and thereby affecting friction and wear processes considerably [2,3].

However, there are many tribo-systems operating in various environments, where such high wear rates or high contact pressures are not relevant. Mechanical components even in conventional engines and machines, are wearing at rates in the order of nanometer per hour, which is a thousand times slower than in many lab tests [4]. Also, more and more applications emerge where sliding occurs over small displacements in the range of micrometers under low normal forces of a few micro/milli-Newtons and low contact pressures of a few MPa or less. Such sliding conditions can be found in micro-motors, bio-implants or MEMS [5].

The selection of appropriate coating materials for such applications is very important in order to reduce not only friction and wear, but also the stiction of the parts. For such applications, the evaluation of materials and coatings by using

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the conventional ‘high load’ pin-on-disk or reciprocating test equipments is not appropriate [6] and the investigation of friction and the evolution of wear at low loads is more suitable and interesting for such applications. The knowledge of tribological phenomena at small scales (microscale) like the occurrence of elastic or plastic deformation, fracture and wear, provides useful information on the basic requirements for achieving the best wear resistance.

Although extensive work was done in the nano- and micro-Newton range with atomic force microscopes [7–9], the milli-Newton range has not yet been explored and explained in detail. The nanoprobe of an AFM simulates a single asperity contact and thereby leads to very high contact pressures, similar to the pressures that exist between the asperities of contacting surfaces in engineering parts. As a result, AFM experiments simulate a single asperity against a surface without any mechanical interlocking between asperities or the distribution of load over multiple asperities as it occurs in many applications. Apart from this, the calibration of an AFM is a difficult task, leading to a fair amount of scatter in the tribological data gathered from it. Moreover, the coefficient of friction of various materials is at the nanoscale quite similar, which makes the evaluation of materials difficult.

This work focuses on wear phenomena in miniature but multiple-asperity contacts using ball-on-flat contact conditions where a mechanical interlocking between the contacting surfaces exist, unlike in wear tests performed with an lateral force microscope. Low load tests were performed with a Falex Modular Universal Surface Tester (MUST) operating in the micro- to milli-Newton range [10]. Hard coatings DLC and TiN were chosen for this study. The influence of a low dissipated frictional energy that is in the range of a few microjoules and of the influence of the coating roughness in the range of a few nanometers, on friction and wear of such coatings, were investigated. An atomic force microscope (AFM) was used to measure the wear morphology at the asperity level and the evolution of the roughness of the coatings with test duration. Surface topography was recorded prior to and after reciprocating sliding tests.

## 2. Experimental

Reciprocating wear tests were performed on coatings at normal forces that are relevant for low load sliding systems. The resulting damage is however so limited that it had to be characterized by AFM. The wear evolution was observed by comparing the coating surface before and after reciprocating tests.

Sliding tests were carried out on commercially standard titanium nitride (TiN) and diamond-like carbon (DLC) coatings. TiN coatings were deposited at WTCM Diepenbeek (Belgium), and Bekaert N.V. (Belgium) supplied DLC coatings. These tests were carried out at low normal forces in the milli-Newton range. A MUST micro-tribometer (Falex Tribology N.V., <http://www.falexint.com>, Belgium) with a

load range from micro-Newton to 10 N was used. Silicon nitride balls of 5 mm diameter were used as counterbody. Coatings and counterbodies were thoroughly cleaned with ethanol prior to experiments. The sliding tests were performed in ambient air at 23 °C and 50% relative humidity. All the tests were carried out at a sliding frequency of 0.2 Hz. In that way very slow and gentle reciprocating sliding tests were achieved.

The tests were done for increasing test durations (number of wear cycles) to observe the evolution of wear. The minimum test duration was chosen in such a way that the wear scars after the tests were visible under an optical microscope in order to position them in the AFM. Care was taken to limit the number of cycles so that the substrate material was not reached during the tests. The surface topography of the coatings was examined prior to the wear tests using a Digital Instruments Nanoscope III Atomic Force Microscope (AFM). The surfaces were scanned with a silicon nitride cantilever with a stiffness of 0.12 N/m [11] both before and after the sliding tests. The thickness of the TiN coatings was between 2 and 3 µm whereas it was 1 µm for the DLC coatings.

For TiN, normal forces of 50 and 200 mN were chosen to compare moderate wear with accelerated wear. For DLC, a slightly higher load of 100 mN had to be chosen because of its good wear resistance. The peak-to-peak displacement amplitude was 400 µm in reciprocating tests on DLC and 300 µm in the case of the TiN coating. All tests were duplicated. A list of wear tests characterized by AFM, is given in Table 1.

AFM scans on the wear tracks were performed before cleaning to investigate the morphology of wear particles and the roughness changes. Then, the wear tracks were cleaned ultrasonically with ethanol. After cleaning the wear tracks were analyzed with AFM to observe the deformation of asperities along with the changes in roughness. The topography of wear tracks was compared with the initial non-deformed surface morphology to understand the wear evolution.

### 2.1. Materials

Mechanical properties like hardness and Young’s modulus of DLC and TiN coatings, and SiN counterbodies are summarized in Table 2. These were measured with a CSM nano-indenter (Berkovich tip) prior to sliding tests.

Table 1  
Overview of test parameters used for reciprocating tests carried out on TiN and DLC coatings using MUST micro-tribometer

Experiment number	Coating	Normal force (mN)	Displacement amplitude (µm)	Number of cycles
1	TiN	50	300	500
2				2000
3		200		50
4				500 1500
5	DLC	100	400	1000
6				3000
7				5000

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