In Operando tribological study at the micro-scale - Application to PVD hard films

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Tribological interfaces characteristics are determined by mechanical stresses and physicochemical conditions in the contact. However, the local evolution of the contact area is mostly unknown. Thus, small-scale dynamic analysis in environmental conditions would allow to well consider phenomena involved between the rubbing materials, leading to a better understanding of the tribological mechanism. Such a local environmental dynamic characterisation does correspond to the *operando* approach. A laboratory-made micro-tribometer was designed and implementable into an environmental SEM or Raman spectrometer. Objective is to analyse, at micro-scale and *in situ*, effect of environment on the tribological behaviour of PVD model hard thin films.

Keywords: micro- tribometer, e-SEM, Raman spectrometer, DLC, TiN

1. Introduction

In-service lifetime of a hard thin film is strongly linked to its tribological behaviour, which in turn is dependent on the nature of the contact. Indeed, if the nature of involved materials, the dynamic of friction, or the macroscopic physicochemical characteristics of the contact can be easily imposed, the real local conditions met in the rubbing zone are difficult to assess. Different strategies were attempted involving transparent counterparts for lubricating contacts [1], or indirect physic measurements of tribofilms (*e.g.* Raman spectroscopy [2]). These approaches gave interesting key-information, but do not consider neither microstructural changes of the stressed surface, nor its direct chemical evolution.
A small-scale analysis under environmental conditions would be required to better analyse the realistic friction behaviour, and predict service life of coated systems. Therefore, a specific ball-on-disk micro-tribometer was designed to provide such local *in operando* measurements in various environments (vacuum, gaz nature and partial pressure, temperature). The different conditions can be controlled thanks to the specific column of the QUATTRO® Thermofischer environmental scanning electron microscope (e-SEM).

2. Methods

The designed reciprocating micro-tribometer is able to acquire measurements under both *ex situ* and *in situ* conditions. In order to test the tribometer itself, and may compare results with the literature, well-known coatings such as TiN (hard film) and a DLC (self-lubricating layer) are deliberately used. The counterface is a steel ball with 10 mm of diameter. The tests are carried out at 1mm/s velocity into the SEM chamber operating under high vacuum, and in humid mode in order to highlight the effect of water. The range of normal load is 6N-30N, leading to 0.5 to 1 GPa Hertzian pressures. The developed "TRIBOMEB" software allows to extract different raw data in terms of normal, lateral and tangential forces, displacement, time, cycle….

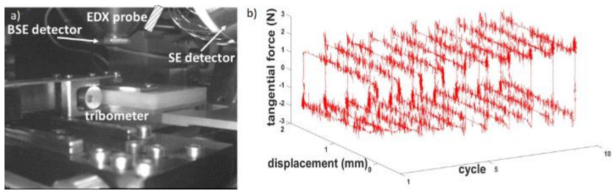

Figure 1: a) Micro-tribometer into e-SEM b) ex. of tangential force versus cycle and displacement (TiN)

3. Results

The friction coefficient is equal to 0.2 (±0.03) for TiN samples, 0.07 to 0.16 for DLC coatings, under ambient conditions and Raman spectrometer. SEM and Raman analysis on DLC coated sample highlight first the formation of a thin layer then a tiny evolution with time at the scale observed. For TiN coatings a continuous formation of submicronic thin third body particles is observed on the track. The tracks on the balls are also analysed at the end of the tests.

In situ tests in SEM provide quite sensitive measurements under *in operando* conditions and micro-scale, which allow temporal and spatial follow-up of tribological characters of various materials.

4. References

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