Surface & Coatings Technology 205 (2010) 1455–1459

Contents lists available at ScienceDirect

Surface & Coatings Technology

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

# In situ accelerated micro-wear — A new technique to fill the measurement gap

T.W. Liskiewicz [a], B.D. Beake [b,*], J.F. Smith [b]

[a] *Institute of Engineering Thermofluids, Surfaces and Interfaces, School of Mechanical Engineering, University of Leeds, Woodhouse Lane, Leeds, LS2 9JT, UK*
[b] *Micro Materials Ltd., Willow House, Ellice Way, Yale Business Village, Wrexham, LL13 7YL, UK*

ARTICLE INFO

Available online 3 August 2010

*Keywords:*
Nanotribology
Amorphous carbon
MEMS
Microtribology
Micro-wear

ABSTRACT

A novel accelerated microtribological capability was implemented on a commercial ultra-low drift nanomechanical test system (NanoTest) by modification of the instrument's hardware. 10 and 25 μm spheroconical and Berkovich diamond probes were used in this study. To compare the accelerated micro-wear capability with existing nano-scratch tests, a range of thin film samples previously characterised were evaluated, including 80 nm ta-C film deposited on Si, 150 nm a-C:H thin film deposited on Si, metal-containing molybdenum disulphide (MoST) 70–150 nm, 70 nm a-C:H and 1 μm a-C films deposited on Si, multilayered 20 nm Si3N4/20 nm NiCr/80 nm Si3N4 multilayer coating deposited on float glass and additionally bulk Cu sample. Operational principles of the experimental setup are explained and reliability of the method is validated with a number of experiments. Results are presented and discussed following four experimental sections of this paper: (i) constant load micro-wear of various films on Si, (ii) constant load micro-wear kinetics of bulk Cu, (iii) ramped load micro-wear of thin films and (iv) tangential force calibration.

## 1. Introduction

Since 1959, when Richard Feyman gave his famous talk “There's Plenty of Room at the Bottom” [1], the nanotechnology concept has been attracting significant interest from the scientific community and the general public. As a result, nanotechnology solutions can be currently found in a number of everyday life products from electronics to medicine [2]. This field has been extensively studied over the last five decades, however there are still many challenges to be addressed and nanotribology is one of many areas contributing to better understanding of phenomena at the nanoscale [3]. Nanotribology research has two main domains associated with (i) fundamental science leading to the understanding of basic principles of friction and (ii) applied science related to technology of small devices like microelectromechanical systems (MEMS), where standard constructs of classical physics do not always hold true.

In the applied nanotribology research area an increasing number of papers with a particular focus on nano- and micro-wear behaviour of materials are being published. Peng et al. studied nano-wear of gold and silver against single silicon crystal using atomic force microscopy (AFM) to perform energy analysis for interfacial reactions [4]. Nano-wear of Sn and Ni–Sn coatings, which can be potentially used for lithium ion battery anodes, was investigated by Chen et al. using nanoindenter and multi-scratch technique [5]. A link has been found here between mechanical degradation of coatings and charge–discharge cycling of batteries. Yu et al. used AFM to study nanofretting behaviour of monocrystalline silicon for potential application in MEMS operating in vacuum conditions [6,7], and it has been observed that there is a different energy ratio related to transition between sliding conditions for nanoscale fretting comparing to classical macroscale fretting. Wilson et al. focused on C and Cr doped amorphous C films being considered as another solution to tribological issues in MEMS devices and characterised these coatings using modified nanoindenter to carry out micro-wear experiments [8–10]. Finally, micro-wear behaviour of DLC and TiN coatings using micro-tribometer under reciprocating sliding was investigated by Achanta et al., where wear mechanism and third body interaction were discussed using AFM analysis [11,12].

Nano- and microscale tribology experiments require specific, high resolution equipment and methods with good stability and ultra-low drift. Reliable, accelerated oscillating micro-wear technique capable of testing under wide range of contact pressures is necessary for applied nanotribology research. Current nano-scratch techniques are limited by unidirectional movement and slow operation [13]. AFM offers a controlled way of performing micro-wear tests under very low loads, however as a piezo-based technology it does not have a necessary stability for long duration tests [14]. Various micro-tribometers are usually based on cantilevers to measure forces, which do not provide enough stiffness to carry out tests under very low displacement oscillations [15,16]. In this paper we present a set of experimental results obtained using a modified nanoindenter platform for accelerated micro-wear testing. Different types of reciprocating micro-wear

---

* Corresponding author. Tel.: +44 1978 261615; fax: +44 1978 356966.
*E-mail address:* ben@micromaterials.co.uk (B.D. Beake).

0257-8972/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.surfcoat.2010.07.109

experiments can be implemented with sample oscillation occurring either under (i) constant applied load or (ii) increasing load. When sample oscillation occurs during a constant load test the evolution of the depth of the wear track under load and time-to-fatigue failure are recorded. When sample oscillation occurs during a slow loading nanoindentation experiment the critical load for film failure and associated critical depth of the wear track under load are recorded. To achieve reliable in situ wear measurements it is necessary that the instrumentation to monitor sub-micron deformation over extended test durations be sufficiently stable to avoid the data being compromised by underlying signal drift. A thermal drift rate of 0.005 nm/s or less is highly desirable as this enables measurements of wear occurring at low contact pressure over periods of 1 h or more.

## 2. Experimental methodology

### 2.1. Modification to a commercial nanoindentation system

The accelerated micro-wear capability has been implemented on a commercial ultra-low drift nanomechanical test system (NanoTest, Micro Materials Ltd). The instrument's hardware was modified to include an additional oscillating stage unit that employs a multilayer piezo stack to generate sample motion. To achieve larger amplitudes, the piezo movement is magnified by means of a lever arrangement as shown in Fig. 1a. The lever is pivoted on a notch hinge that rotates elastically. The sample holder is held in a stiff spring flexure assembly providing a restoring force without backlash.

Loading and sample movement are controlled independently in the instrument software so that the hardware modification to enable sample oscillation does not require significant software alteration and the loading and contact routines of the commercial nanoindentation module can be used. The sinusoidal piezo driving voltage is produced by a signal generator + amplifier + transformer combination.

The track length is determined by a separate calibration procedure that enables the voltage applied to the piezo stack to be correlated to the length of the wear track produced (Fig. 1b). To do this, the oscillation is applied directly to the diamond holder such that the displacement can be monitored via the instrument's capacitive sensor. Since the micro-wear stage displacement occurs perpendicularly to the test probe axis, this calibration requires that the micro-wear stage be temporarily mounted as shown in Fig. 1c.

10 and 25 μm spheroconical and Berkovich diamond probes were used in this study. The oscillation frequency was 20 Hz unless otherwise mentioned. For the figures, the data has been filtered (6-point moving average) to remove aliasing effects and enable the failure events to be more clearly delineated. All data are shown as probe depth under load rather than residual (wear) depth after removal of load.

### 2.2. Samples

To compare and contrast the accelerated micro-wear capability with existing ramped and repetitive constant load nano-scratch tests, a range of thin film samples previously characterised extensively by nanoindentation and nano-scratch testing [18] have been evaluated. The 80 nm ta-C film was deposited on Si using the FCVA process in an industrial filtered cathodic vacuum arc system (Nanofilm Technologies Pte. Ltd., Singapore) evacuated to a base pressure lower than $1\times10^{-6}$ Torr. The 150 nm a-C:H thin film was deposited on Si (Western Digital, Thailand). Metal-containing molybdenum disulphide (MoST) 70–150 nm, 70 nm a-C:H and 1 μm a-C films were deposited on Si wafers using a closed field un-balanced magnetron sputtering ion plating technique (Teer Coatings, UK). The $R_a$ roughness of the MoST, a-C:H, and ta-C films was 0.5–0.7 nm; for the 1 μm a-C film it was 4 nm. The multilayered coating was an experimental sample (Guardian Inc., USA) with a nominal structure 20 nm Si3N4/20 nm NiCr/80 nm Si3N4 multilayer deposited on float glass. Additionally, the Hard Drawn High Conductivity copper (HDHC, 99.9 wt.% Cu) was set in resin and metallographically polished to a smooth surface finish.

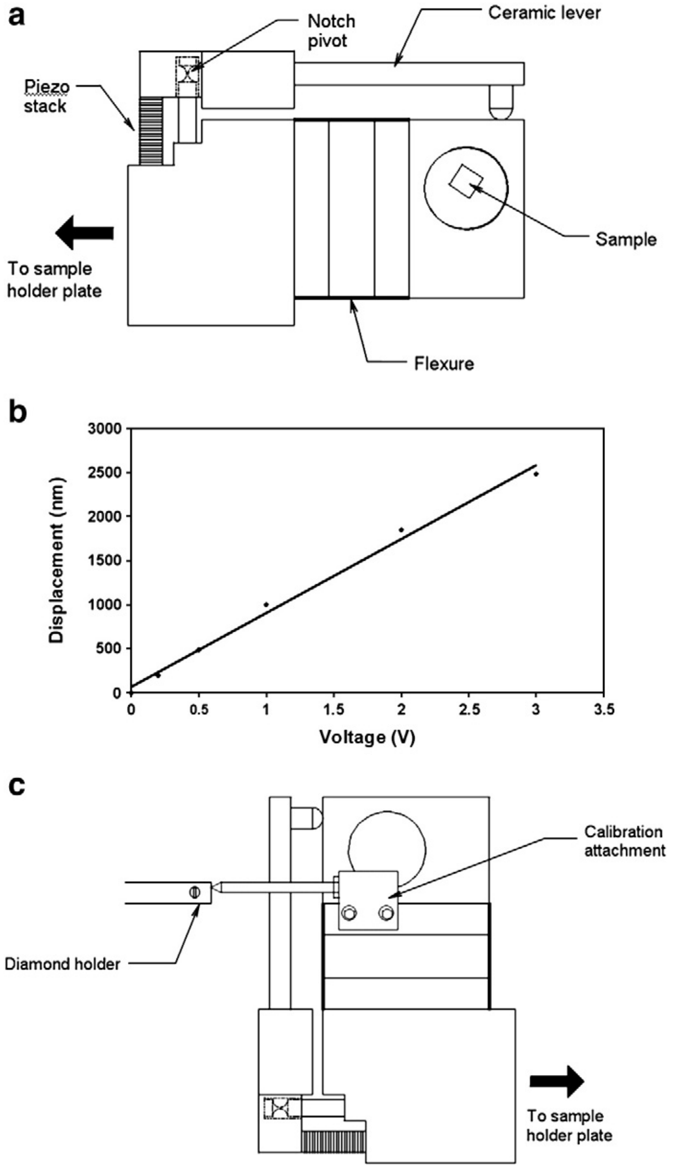

**Fig. 1.** (a) Schematic representation the experimental configuration for accelerated micro-wear; (b) typical track length calibration output; measured displacement (nm) as a function of applied voltage (V); (c) configuration for track length calibration.

## 3. Results

### 3.1. Constant load micro-wear of 70–150 nm thin films

Comparative behaviour for micro-wear of various sub-200 nm coatings deposited on Si with a 25 μm spheroconical probe is shown at 10 mN in Fig. 2. During the duration of the 10,000 cycle test no abrupt film failure events were observed but it is evident that the evolution of the probe depth under load of the thin coatings varies considerably, with the 80 nm ta-C being the most resistant and the 70 nm MoST being the least. One of these samples, the 70 nm a-C:H film, was selected for further study and its micro-wear behaviour as a function of applied load (0.2–100 mN) is shown in Fig. 3. No abrupt changes in probe depth were observed over the load range studied suggesting a gradual wear process with the absence of discrete fatigue fracture events.