Wear 301 (2013) 575–582

Contents lists available at SciVerse ScienceDirect

Wear

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

Nano-scratch, nanoindentation and fretting tests of 5–80 nm ta-C films on Si(1 0 0)

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ARTICLE INFO

Article history:
Received 19 September 2012
Received in revised form
25 January 2013
Accepted 28 January 2013
Available online 4 February 2013

Keywords:
Nanotribology
Nano-fretting
ta-C
Nanomechanics
MEMS

ABSTRACT

Wear and stiction forces limit the reliability of Silicon-based micro-systems when mechanical contact occurs. Ultra-thin filtered cathodic vacuum arc (FCVA) ta-C films are being considered as protective overcoats for Si-based MEMS devices. Fretting, nano-scratch and nanoindentation of different thickness (5, 20 and 80 nm) ta-C films deposited on Si(1 0 0) have been performed using spherical indenters to investigate the role of film thickness, tangential loading, contact pressure and deformation mechanism in the different contact situations. The influence of the mechanical properties and phase transformation behaviour of the silicon substrate in determining the tribological performance (critical loads, damage mechanism) of the ta-C film coated samples has been evaluated by comparison with previously published data on uncoated Silicon. The small scale fretting wear occurs at significantly lower contact pressure than is required for plastic deformation and phase transformation in nanoindentation and nano-scratch testing. There is a clear correlation between the fretting and nano-scratch test results despite the differences in contact pressure and failure mechanism in the two tests. In both cases increasing film thickness provides more load support and protection of the Si substrate. Thinner films offer significantly less protection, failing at lower load in the scratch test and more rapidly and/or at lower load in the fretting test.

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1. Introduction

Wear and stiction forces limit the reliability of Silicon-based micro-systems (e.g. MEMS) when mechanical contact occurs [1–3]. Silicon exhibits highly complex mechanical and tribological behaviour with phase transformations and lateral cracking observed in indentation and brittle fracture in a wide range of mechanical contacts [4–6]. It exhibits little or no conventional plasticity at room temperature and its deformation is dominated by phase transformation and fracture. Its nanoindentation behaviour has been extensively studied [7–12]. It has been established that a phase transformation to metallic behaviour occurs beneath the contact site and the pop-out during unloading is a consequence of phase transformation and volumetric expansion. Less well studied is its behaviour under the type of more complex loading geometries that can occur in practical tribological situations [13] so we recently investigated the behaviour of highly polished Si(1 0 0) under different contact situations, using a nanomechanical test system to perform nanoindentation, nano-scratch and 10,000 cycle small scale fretting tests all with the same 4.6 μm sphero-conical diamond indenter [14,15]. Tangential loading in the nano-scratch and fretting tests promoted non-elastic yield at lower critical load. Silicon showed subtle rate sensitivity in the nano-scratch and nanoindentation tests but the wear behaviour in the fretting test was more strongly dependent on the rate of initial loading. When the load was applied abruptly in < 0.3 s, radial and lateral cracking and material removal was observed and large displacement jumps (pop-ins) were observed during the subsequent fretting test. In contrast, when the load was applied more slowly in 10 s radial cracking was not observed and there was a distinct threshold load at around 100 mN marking the transition to a more severe wear mode with extensive lateral cracking and material removal.

Several approaches are being considered including liquid lubrication [16], solid lubrication with self-assembled monolayers [17], ALD [18,19] and carbon films [20–23]. Particularly promising results on actual MEMS devices have been obtained for conformal deposition strategies (WS_2 by ALD [19], DLC by PECVD [20]). Tetrahedral amorphous carbon (ta-C) films deposited by FCVA have been developed for MEMS applications including capacitive sensors and protective coatings for micromachined components [21–23]. The mechanical and interfacial behaviour of the contacting silicon

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0043-1648/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.wear.2013.01.073

surfaces is modified by these thin, low surface energy films. Nanoindentation with a Berkovich indenter has shown that the films are hard (hardness of 80 nm ta-C is ~22 GPa) and elastic [24,25]. This high hardness and H/E is due to >70% sp^3 [26] but the films can be highly stressed if too thick [27,28]. Nano-scratch testing of ta-C films has shown that they are sufficiently thin to not show large area delamination and scratch resistance increases with H/E [24,25]. Increasing the ta-C thickness from 5 to 80 nm was found to increase the critical load for film delamination by a factor of two.

In this current study nanoindentation and nano-scratch tests have been performed on 5, 20 and 80 nm ta-C films with a 4.6 μm radius probe, and fretting with 5 and 37 μm probes to determine (i) rate sensitivity of pop-outs during unloading in nanoindentation (ii) whether the thin ta-C films influence the occurrence of large pop-ins at high load in nanoindentation (iii) the extent of rate sensitivity in film failure in the nano-scratch test (iv) the influence of the ta-C on lateral cracking thresholds in nano-scratch testing (v) the mechanism of film failure in the fretting test and the influence of film thickness on the fretting wear. The influence of the mechanical properties and phase transformation behaviour of the silicon substrate in determining the tribological performance (critical loads, damage mechanisms) of the ta-C film coated samples has been investigated by comparison with previously published [14,15] data on uncoated Silicon acquired with the same probe and experimental conditions.

2. Experimental

Nanoindentation and nano-scratch tests were performed on ta-C films deposited on silicon wafers with a commercial ultra-low drift nanomechanical test system (NanoTest, Micro Materials Ltd.) fitted with a 4.6 μm end radius sphero-conical diamond indenter which was calibrated by nanoindentation measurements on a fused silica reference sample over a wide load range. ta-C films of 5, 20 and 80 nm thickness were deposited on Si(1 0 0) substrates by FCVA [24,25]. The substrates were ultrasonically cleaned with deionized water for 10 min, followed by drying with a static neutralizing blow off gun. The samples were placed in the deposition chamber of an industrial filtered cathodic vacuum arc system (Nanofilm Technologies Pte. Ltd.) evacuated to a base pressure lower than 1×10^{-6} Torr. Prior to deposition, the silicon surface was sputtered by an argon ion beam from a dc ion beam source for 3 min to remove the native oxide. The substrate holder was in floating bias. The film thickness of the 20–80 nm films was measured by a surface profiler and the thickness of the 5 nm film was estimated from the deposition rate. The R_a surface roughness was 1.8, 2.1 and 3.1 nm for the 5, 20 and 80 nm films respectively [24]. The influence of the films on the composite film-substrate response was investigated by comparing results to those on an uncoated highly polished 300 μm thick Si(1 0 0) wafer provided by PI-KEM (Tamworth, UK) using the same 4.6 μm sphero-conical diamond indenter.

Three repeat indentation tests to 10, 50, 100, 200, 300, 400, 500 mN peak load, loading and unloading in 20 s, with a 5 s hold at peak load were performed on the 5, 20 and 80 nm films. Further tests were performed on the 80 nm ta-C to a range of peak loads and unloading rates with a 5 s hold at peak load: (i) Peak load=100 mN, loading rate=5 mN/s; unloading rate=1–50 mN/s, 10 repeats for each unloading rate; (ii) Peak load=200 mN, loading rate=10 mN/s; unloading rate=1–50 mN/s, 20 repeats for each unloading rate. In all tests a 60 s hold at 90% unloading was used to correct the data for (any) thermal drift.

Nano-scratch tests to 200 mN were performed on the 80 nm ta-C film tests over a wide range of loading rates (dL/dt=0.1–12 mN/s) and scan speeds (dx/dt=0.1–40 μm/s) to provide critical load data over a range of dL/dx=0.1–100 mN/μm. One to five repeat tests were performed for each set (11 in total) of experimental conditions. Tests were performed as multi-pass (3-scan) experiments (topography-scratch-topography) that were subsequently analysed in the NanoTest software to determine the on-load and residual depth data, following the procedure described in Ref. [18]. Additional scratch tests were carried out on the ta-C films and uncoated Si(1 0 0) to a peak load of 300 mN with a loading rate of 12 mN/s and scanning speed 10 μm/s to investigate lateral cracking (three repeats on Si, five on the ta-C films). All tests were spaced at least 50 μm apart.

The fretting tests were performed in a NanoTest Vantage system using 5 and 37 μm diamond probes whose end radii were calibrated by nanoindentation testing over a wide load range on fused silica and sapphire (0 0 0 1) reference samples. The configuration for small scale fretting includes an additional oscillating stage with a multi-layer piezo-stack to generate sample motion. The piezo movement is magnified by means of a lever arrangement to achieve larger amplitudes [29]. The fretting track length was set at 10 μm and the oscillation frequency 5 Hz. Fretting experiments of differing number of wear cycles and applied load were performed in normal laboratory conditions (~50% RH) on the ta-C films as summarised in Table 1. The mean initial Hertzian contact pressures in the tests ranged from ~3–4 GPa at 10 mN to ~10 GPa at 200 mN using the 37 μm probe and were ~6 GPa using the 5 μm probe. Adjacent tests were spaced 100 μm apart. Tangential (friction) force data were acquired simultaneously with depth data throughout the nano-scratch and fretting tests using a lateral force transducer which was calibrated by a method of hanging masses.

The Electron microscopy was done utilising Zeiss Supra 40 VP (variable pressure) electron microscope. Secondary electron imaging was done by conventional (Everhart-Thornley) and In-Lens secondary electron detector. The detectors have different sensitivity to the surface charge and topography, when In-Lens detector is more sensitive to surface charging and less sensitive to topography. Surface charging can be caused by the surface

Table 1
Fretting test experimental conditions.

	5 nm ta-C	20 nm ta-C	80 nm ta-C
R=37 μm, L=1 mN		900 cycles	
R=37 μm, L=10 mN	300, 600, 900, 1500, 3000, 6000, 9000, 18,000 cycles	1500, 3000, 6000, 9000, 18,000 cycles	
R=37 μm, L=50 mN		300, 600, 900, 1500, 3000, 6000, 9000 cycles	1500, 3000, 6000, 9000, 18,000 cycles
R=37 μm, L=200 mN			300, 600, 900, 1500, 3000, 6000, 9000, 18,000 cycles
R=5 μm, L=10 mN			1500, 3000, 6000, 9000, 18,000 cycles

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