Surface & Coatings Technology 203 (2008) 794–798

Contents lists available at ScienceDirect

Surface & Coatings Technology

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

Nano-lamination in amorphous carbon for tailored coating in micro-dry stamping of AISI-304 stainless steel sheets

Tatsuhiko Aizawa [a,*], Eiji Iwamura [b], Kuniyoshi Itoh [c]

[a] AsiaSEED-Institute, 3-15-10 Minami-Rokugo, Ota-city, Tokyo 144-0045, Japan
[b] R & D Center, Arakawa Chemical Industry, Ltd., Osaka 538-0053, Japan
[c] Seki-Corporation, Col. Ltd., Tokyo 192-0046, Japan

ARTICLE INFO

Available online 4 September 2008

Keywords:
Amorphous carbon
Nanolamination
Mechanical properties
Micro-dry stamping
WC tool
AISI-304 stainless steel sheets

ABSTRACT

Nano-lamination is a new way to make full use of multi-layered structure for coating instead of the mono-layered coating system. Different from the conventional nano-lamination approach, where two different kinds of material system are deposited in layers, the amorphous carbon layer, a-C:H, is alternatively deposited with graphite-like cluster layer, resulting in an amorphous carbon base nano-laminated coating. In this nano-lamination, the pulse bias voltage and the pulse duration time are programmed to control the bi-layer thickness and number of laminates. Furthermore, the mechanical properties are also controllable by varying the bi-layer thickness and sub-layer ratio. In this paper, the effect of number of layers and bi-layer thickness on the mechanical properties is investigated by the nano-indentation technique. Higher hardness and Young's modulus is attained with reduction of bi-layer thickness. The scratching test of this nano-laminated coating is made to demonstrate that it has sufficient scratch load above 100 N. Furthermore, a dry micro stamping test is performed to prove that this nano-laminated coating has sufficient wear-toughness enough to make dry stamping in 10,000 times in practice even if it has nearly the same Young's modulus and hardness as the mono-layered coating. No delamination or break-away occurs on the ironed surface of coated tools while severe delamination is observed in the conventional mono-layered coating.

1. Introduction

Environmental archiving in manufacturing requires significant reduction of burdens and less emission of wastes [1]. Although lubricating oils have been used to reduce the friction and wear of tools and dies in stamping, their post-treatment has a risk of air and water pollutions and becomes an issue of nuisance in cost competitiveness. Then, a trend toward the lubricating oil free or dry forming has become a main stream as an environmentally benign manufacturing [2]. In particular, lubricating oils and cleansing agents are completely disliked in the fine, precise stamping of electrical parts since their residues often lead to deterioration of quality in products [3]. Then, multi-step progressive stamping aims to be lubricating-oil free or dry without loss of geometric accuracy [4]. Various protective coating technologies have been developed and applied to realize dry stamping [5]. DLC (diamond like carbon) and diamond coatings are a typical method to reduce the friction and wear of punches and dies in stamping by their high hardness [6]. This hard coating approach is not always suitable to dry micro stamping, requiring the narrow clearance and thin coating layer. Self-lubricating coating by MoS_2/WS_2 is also employed to reduce the friction coefficient and specific wear volume [7]. Since solid-lubricating debris particles are ejected from coating films, fine clearance between tools and work materials has a risk of blockage to terminate the dry stamping process. Authors have developed the nano-structured a-C:H coatings for dry stamping processes [8–11]. In particular, nano-lamination is preferable to dry micro stamping since the mechanical properties of coating are tailored to each requirement in its progressive steps.

Two different kinds of metallic alloys and ceramic compounds are alternatively deposited to form a nano-laminated coating film; e.g. TiN/Ti–B–N [12] and TiN/TiAlN [13]. In our developing nano-laminated DLC coating, the higher density amorphous carbon layer is alternatively stacked with the lower density graphitic carbon layer to form a bi-layer structure and to tailor its hardness for each usage of tools and dies in dry stamping [10–11,14]. Owing to this hard/soft nano-lamination with density distribution, original mechanical properties to amorphous carbon coating, is reinforced by ductility of graphitic layers.

In the present paper, this nano-laminated a-C:H coating is much investigated toward tailored coating for dry fine and micro progressive stamping. First, nano-lamination via unbalanced magnetron sputtering is described by high resolution in-lens SEM. Mechanical properties of this nano-laminated a-C:H coating is studied by the scratching test and nano-indentation methods. Nano-lamination effect on the mechanical properties is further discussed by varying

* Corresponding author. Tel.: +81 3 5480 0343; fax: +81 3 5480 0344.
E-mail address: aizawa@asiaseed.org (T. Aizawa).

0257-8972/$ – see front matter
doi:10.1016/j.surfcoat.2008.08.059

the number of laminated layers and the bi-layer thickness. Finally, the fine micro-stamping apparatus is used to demonstrate the integrity and wear toughness of nano-laminated a-C:H coating on WC tools.

2. Experimental procedure

2.1. Coating method

Un-balanced magnetron sputtering was used for film deposition of amorphous carbon (a-C:H) by using the carbon target with the purity of 5 N. The sputtering pressure was constant, P=0.5 Pa; the gas mass-flow ratio of CH_4 to Ar was also constant, 15%. Both mono-layered and nano-laminated a-C:H films were prepared with use of pulse bias voltage sequence. The bias voltage (V_b) to form mono-layered films was constant, V_b=−200 V. While, V_b was alternatively controlled, to be 0 V for formation of lower density graphitic layers and to be −200 V for formation of higher density disordered layers. The applied power was constant, 1 kW. The pulse duration ($\Delta\tau$) was preset to be $\Delta\tau=(t_F/2L)/D_p$, where t_F is the total film thickness, L, the number of laminates, and D_p, the deposition rate. The measured D_p in this coating was around 7 nm/min.

Silicon substrate with 6 N purity and 20×20×0.5 mm^3 was utilized for microstructure observation and nano-indentation tests; t_F=250 nm. A cemented carbide (WC) circular disc with the diameter of 10 mm was employed for scratch test; t_F=1 μm. WC upper punch with the curved head of 2.1 mm in width and 1.2 mm in thickness, was also prepared for bending and ironing process in the dry micro-stamping test; t_F=1 μm. The graded interlayer was first deposited with the thickness of 50 nm.

2.2. Microstructure characterization

The field-emission type, in-lens SEM, S-4800 (Hitachi) with the resolution of 1 nm by 15 kV, was used for microstructure observation both on the surface and in the cross-section of coated silicon substrate. A sample for cross-sectional observation was prepared by cracking the brittle substrate, and, the fracture surface was set up in the sample-holder. Electron beam intensity was controlled to be weak enough to reduce the irradiation damage on the fracture surface.

2.3. Mechanical measurement

Tribo-indentation system ENT-1100a (Hysitron Inc.) with the Berkovich type diamond indentor, was used for nano-indentation testing. In this case, the indentation depth is controlled to be less than 1/6 of total thickness, in order to be free from the effect of substrate on the load-displacement curve. Both the Young's modulus and hardness were measured as the average of five sampling data. The scratching tester equipped with acoustic emission sensor (REVETEST, CSEM, Co. Ltd.) was used with the lateral speed of 10 mm/min, the loading rate of 100 N/min and the maximum load of 100 N. The surface profilometer (ZYGO) was utilized to measure the geometry and dimension around the scratched trace.

2.4. Dry micro stamping method

A micro-stamping test machine was used to run the progressive stamping with the maximum load of 5 kN and the working speed of 60 rpm. AISI-304 austinitic stainless steel sheet with the thickness of 0.211 mm was employed as a work material. In the step of shearing process, holes were punched out without any significant burrs only by using the bare cemented carbide tools and dies. In the bending and ironing step, severe wear might occur on the ironed surface between punch and work sheet; no lubricating oils was never used since the total clearance between die and punch is strictly fixed to be less than 2 to 3 μm. In fact, mono-layered DLC coated tools suffer from severe delamination even after punching out by 100 times, and, the severe galling of stainless steel sheet occurs on the uncoated WC tools after 200 stamping operations [11].

In the present stamping experiment, the number of operations (N) was increased step-by-step up to N=10000. The thickness and surface roughness was evaluated by fine micrometer with the resolution of 1 μm and surface profilometer. In addition, the surfaces of coated punch were also observed by optical microscope.

3. Experimental results

3.1. Microstructure

In the present nano-lamination coating process, each sublayer was deposited with the same deposition rate. Hence, the bi-layer thickness ΔL is designed by $\Delta L_{design}=D_p \times \Delta\tau$. Fig. 1 a) shows a typical cross-sectional view of the nano-laminated coating film. As suggested by previous work [10], the white layer or high density layer, corresponds to a disordered amorphous carbon sub-layer (a-C:H) while the black layer or low density layer, a graphite-like carbon layer. The total thickness (t_F=250 nm) is composed of 13 layers (L=13); $\Delta\tau_1$=160 s and ΔL_{design}=19 nm. The measured ΔL in Fig. 1 a) is 17–21 nm. This agreement of bi-layer thickness assures that nano-lamination process should be accurately controlled by pulse bias voltage sequence. Fig. 1 b) depicts the cross-sectional view of nano-laminated coating when $\Delta\tau_2$ was controlled to be half of $\Delta\tau_1$ for Fig. 1 a): L=25, $\Delta\tau_2$=80 s and ΔL_{design}=10 nm. ΔL in Fig. 1 b) was 9 to 11 nm; bi-layer thickness is

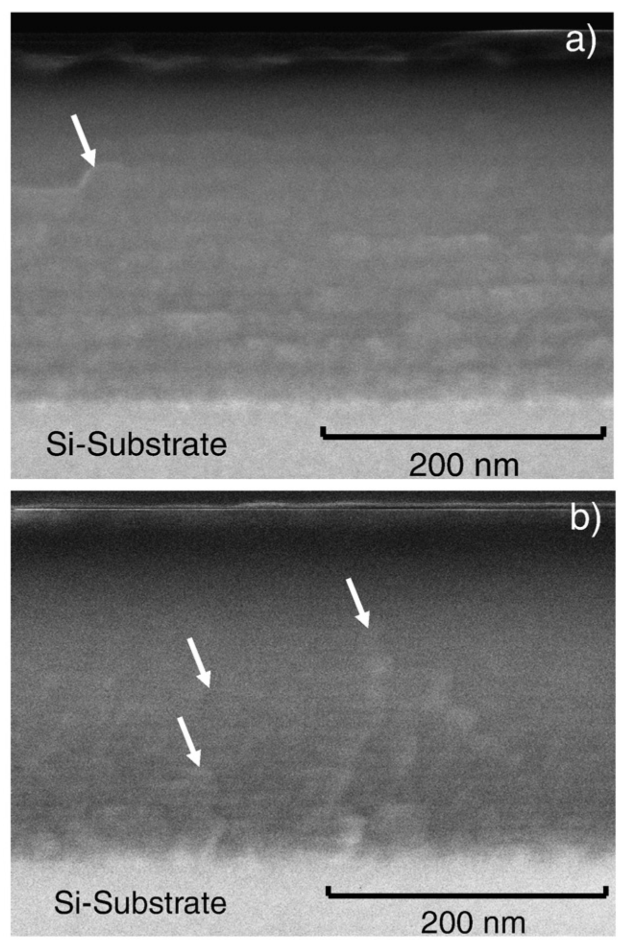

Fig. 1. Scanning electron micrograph of cross-sectional fractured surface of nano-laminated amorphous carbon films: a) L=13, ΔL_{design}=19.nm, and b) L=25, ΔL_{design}=10 nm.