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Nanomechanical properties through nanoindentation method of amorphous carbon and carbon nitride films synthesized by shielded arc ion plating

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Abstract

Amorphous carbon (a-C) and carbon nitride (a-CNx) films were deposited by means of shielded arc ion plating (SAIP) with an arc current of 60 A operated in a gas pressure of 1 Pa. A bias voltage in the range from 0 to −500 V was applied to a substrate during film deposition. Nanomechanical properties of the films were measured by a nanoindentation interfaced with an atomic force microscopy (AFM) using a diamond tip. The nanoindentation was also applied to evaluate wear resistant behavior of the films in nm scale. The a-C film prepared at a substrate bias voltage (V_b) of −100 V was hardest in the present study so as to show a hardness of 43±3 GPa. This a-C film was most wear resistant as well. The a-CNx film prepared at V_b of −300 V possessed the maximum hardness of 14±1 GPa among the prepared a-CNx films. Independently of V_b, all of the a-CNx films showed better wear-resistance characteristics than sapphire and quartz. Although the wear-resistance of the films was not directly correlated to its hardness, elastic modulus, elastic recovery, plastic deformation energy, these properties were certainly govern the wear-resistance of the film.
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Keywords: a-C film; a-CNx film; Shielded arc ion plating (SAIP); Nanoindentaion; Wear-resistance

1. Introduction

Diamond-like carbon (DLC) has been attracted great attention as a hard material for wear resistant applications. In addition, carbon nitride is also attractive for tribological applications, of which the crystal form of β-C_3N_4 has expected to show an elastic modulus hypothetically comparable to diamond. However, thin films of both materials synthesized to date contained large fractions of amorphous structure. Nevertheless, such amorphous carbon (a-C) and carbon nitride (a-CNx) films are promising for many industrial applications owing to their high hardness, wear-resistance and solid lubricity [1–3]. Furthermore, these films recently attract much attention as biomedical materials, since the films have chemical and mechanical durabilites in vivo [4]. To satisfy requirements in high technological applications such as protective coatings of artificial joints like hip joints for biomedical implants, magnetic hard disk, and microelectromechanical systems, many researches have been studied using various deposition methods, for examples, chemical vapor deposition (CVD), sputtering, ion beam deposition, laser ablation and arc plasma deposition [5–10]. Although mechanical and tribological properties of a-C and a-CNx films have been frequently reported, the mechanical properties were discussed mainly from a viewpoint of their unique deposition condition and intensively studied in relation to chemical structures of the films. In particular, the properties of a-CNx are great interest since the a-CNx films showed an excellent wear-resistance, even though its hardness is not so high in comparison with a-C [11,12].

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In this work, a-C and a-CNx films are deposited by shielded arc ion plating (SAIP). A nanoindenter interfaced with an atomic force microscope (AFM) using a diamond tip was used to measure mechanical properties of the films including hardness, elastic modulus, recovery, contact stiffness and plastic and elastic deformation energies as well as to evaluate wear resistant behavior of the films in nm scale. The wear resistant behavior is discussed in relation to other mechanical properties of the films.

2. Experimental

The a-C and a-CNx films were deposited on n-type silicon (100) substrates by the SAIP system (Nissin electric) using a high purity sintered graphite target (Toyo Tanso IG510, ash 10 ppm) as a carbon source. The substrate was located 210 mm from the target. A shielding plate was inserted between the target and substrate. A detailed description about the apparatus was reported elsewhere [13]. In this study, in order to increase deposition rate, the distance between the substrate and shield plate was set to be 50 mm which was 10 mm longer than before. The silicon substrates were ultrasonically cleaned in acetone and ethanol for 20 min in that order. The chamber was evacuated down to a pressure of 2.3×10^{-3} Pa prior to introducing a reaction gas, that is, argon or nitrogen gas with a purity of 99.999% for preparing a-C or a-CNx films, respectively. Each gas introduced into the vacuum chamber through a mass flow controller. To remove contamination and oxide layers on the substrate surface, ion bombardment cleaning was carried out for 10 min in argon or nitrogen plasma at a gas pressure of 10 Pa with a substrate bias (V_b) of −700 V. The films were prepared using Ar or N_2 arc plasma with a DC arc current of 60 A by applying a V_b in the range from 0 to −500 V. The gas pressure during deposition was fixed at 1 Pa. Thickness of the films were adjusted 120±10 nm by controlling the deposition time. Nanohardness and wear-resistance of the films were measured using a nanoindentation (Hysitron, TriboScope) interfaced with an atomic force microscope (AFM, JEOL, JSPM-4210). An identical diamond tip (Berkovich type: 65.3° of half angle) was used throughout in this study. A force-displacement curve of each film was measured with a peak load force ranging from 250 to 2000 μN. Loading and unload times were set both to be 5 s. A typical load-displacement curve is shown in Fig. 1. A displacement, that is, the difference between the loading and unloading curves, was obtained. The hardness was calculated from the unloading curve using the relation with contact area and maximum load. Elastic and plastic deformations occurred on the surface of sample as the indenter pressed into the sample. The areas of OPh_f and h_fPh_c correspond to the plastic and elastic deformation energies, respectively. A wear depth of each sample, to which the diamond

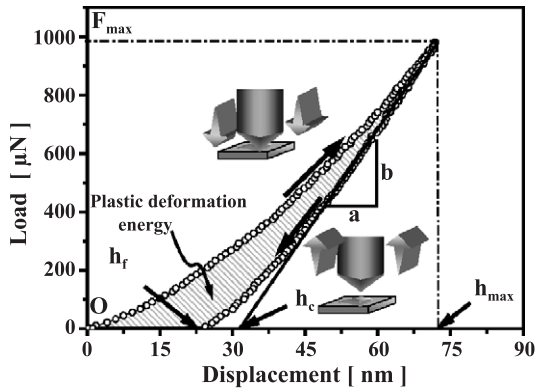

Fig. 1. Typical load-displacement curve with applying force of 1000 μN on the quartz surface.

pyramidal tip scanning at a constant load force of 20 μN had been repeated 20 cycles in 1 μm^2 area at a scanning rate of 2.8 μm/s, was used as an indicator of wear-resistance. In particular, the wear resistant behavior of a-C and a-CNx films deposited at a substrate bias voltage of −100 V was further measured by changing the scanning rates (1.8, 2.8, 5.6 μm/s) and loads (20, 30, 40 μN).

3. Results and discussion

Elastic modulus and hardness of the a-C and a-CNx films are shown in Fig. 2 as well as those of sapphire, fused quartz, and single crystal silicon which are elastically homogenous with the indent depth and crystal orientation. The obtained elastic moduli of these reference samples were 350±25, 69.5±1 and 155±5 GPa and their hardness were 35±5, 9.0±0.5 and 10.2±0.5 GPa, respectively. Results of variations in elastic modulus and hardness in accordance with increasing loads might be affected by indenter tip rounding and a deformation of indenter during indentation or tip blunting. The effects of indenter tip rounding and deformation of indenter are well described by Gong et al. [14]. In addition, Lemoine et al. [15] fairly took into account the effects of tip blunting on tetrahedral amorphous carbon and hydrogenated amorphous carbon thin films. These effects are not discussed in detail in this study. To obtain exact hardness of a film without affected with its substrate, Oliver et al. [16] suggested that each indentation depth had to remain less than 10 % of the film thickness. In contrast, Boyer [17] recommended an indentation depth less than 20 % of the film thickness to avoid the substrate effect. The maximum indentation depth (h)/film thickness (t_f) of films varied from 10% to 30% at the maximum load of 500 μN according to their hardness. Presented the values of elastic modulus and hardness for the films were based on measurement of a load of 500 μN. At this time, the penetration depths were extremely small with below 60 nm that the hardness showed lowest value. At penetration depth less than 50 nm, indenter tip rounding

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