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Abstract

Hard, adherent, and low-friction silicon-containing diamond-like carbon coatings (Si-DLC) have been synthesized at room temperature by 40 keV ($N^+ + N_2^+$), 50% Ar^+/50% ($N^+ + N_2^+$), and Ar^+ ion-beam-assisted deposition (IBAD) of a tetraphenyl-tetramethyl-trisiloxane oil on silicon and sapphire substrates. X-ray diffraction analysis indicated that all coatings were amorphous. The average coating wear rate and the average unlubricated steel ball-on-disk friction coefficient, μ, decreased with increasing fraction of nitrogen in the ion beam, along with an increase in the average coating growth rate. The Knoop microhardness and nanohardness values of the coatings synthesized by the mixed argon and nitrogen ion beam were higher than the values for the coatings synthesized with 100% nitrogen or 100% argon ion beams. These friction/wear improvements are tentatively attributed to both increased hardening due to greater penetration and ionization induced hardening by the lighter (N) ions and to the presence of SiO_2 on the surface of N-bombarded samples.

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1. Introduction

Films of many promising tribological materials, including conventional diamond-like carbon (DLC), have been successfully deposited by ion-beam-assisted deposition (IBAD). The friction coefficient of unlubricated DLC films in dry gases can be as low as 0.01, but this value can reach values as high as 0.10 and 0.20 when measured in a 10% relative humidity [1–3]. However, various researchers have shown [2–4] that DLC films containing elements such as silicon (Si) and titanium (Ti) retain low pin-on-disk friction coefficients in humid environments. DLC films containing silicon (Si-DLC) exhibit friction coefficients as low as 0.04 [2–4] at ambient humidity and temperature and are therefore highly promising for tribological applications. Several trial industrial applications of DLC, including protective wear coatings on bearings and forming tools [5], are limited because of the poor thermal stability of DLC above 350° C. However, there are data [5, 6] indicating that the presence of additional elements (Fluorine [F], Si, and nitrogen [N]) in the coating can increase the range of the thermal structural stability of DLC by as much as 100° C.

In this report, we will discuss the properties (stoichiometry, thickness, microhardness, bonding, adhesion, friction, and wear) of IBAD Si-DLC coatings synthesized with nitrogen ion beams (N/Si-DLC), argon plus nitrogen ion beams ((Ar + N)/Si-DLC), and argon ion beams (Ar/Si-DLC).

2. Experimental Details

A ZYMET 100 nonmass analyzed ion implanter was used for the synthesis of Si-DLC coatings using energetic 40-keV ion bombardment of a vapor-deposited tetraphenyl-tetramethyl-trisiloxane (Dow Corning 704) diffusion pump oil. The nitrogen beam consisted of a mixture of roughly 40% N^+ and 60% N_2^+, yielding about 1.6 nitrogen atoms per unit charge. The diffusion pump oil precursor was evaporated from a heated (145° C) copper oil container through a 3-mm-diameter, 2-mm-thick aperture. The substrates, silicon and sapphire (for Raman analysis only), were initially cleaned in methanol and acetone and then sputter-cleaned with a 40-keV ion beam (10 $\mu A/cm^2$) of the aforementioned gaseous species for 10 min. The temperature of the substrate was maintained close to room temperature using heat-conducting vacuum grease to hold the sample on a water-cooled sample stage. The substrate was inclined at 45° with respect to both the horizontal ion beam and the vertical flow direction of the vaporized oil. The aperture to substrate distance was 0.15 m with a shutter placed above the oil container to start and stop the oil deposition. The growing film surface was continuously bombarded by the aforementioned various ion beams at 40 keV. The base pressure was 2.66×10^{-4} Pa (2×10^{-6} Torr), and the deposition was carried out at 4×10^{-3} Pa (3×10^{-5} Torr) pressure, as in previous work [4]. All coating deposition runs lasted 100 min, resulting in coating thicknesses ranging from 420 nm (Ar/Si-DLC), 505 nm ((Ar + N)/Si-DLC), and 714 nm (N/Si-DLC).

The thicknesses of the films were measured with the aid of a profilometer. The microhardness values of the coatings were measured using a Knoop microhardness tester with a 0.15-N load. The nanohardness values of the coatings were measured with a Nano Instruments XP nanoindenter using an average load of 20 mN [7]. A ball-on-disk tribometer with a 1.27-cm (1/2 in) diameter AISI 52100 alloy steel ball under a 0.5-N load was used to determine the unlubricated sliding friction coefficient μ. Rutherford backscattering spectrometry (RBS) was performed on the films using a 2-MeV He^+ beam and a backscattering angle of 170° to determine their near-surface elemental composition using the RBS simulation program RUMP [8]. An APD (automated power diffractometer) 1710 System 1 was used to study the crystallinity of the coating. Cu $K_{\alpha 1}$ and $K_{\alpha 2}$ x-ray, 0.154060-nm and 0.154439-nm wavelength, respectively, were used for the x-ray analysis for diffraction angles 2θ from 15 to 65° at a scan rate of 0.020°/s.

3. Results and Discussion

3.1 Coating Appearance. Optical microscopy showed that while there were pinholes in the Ar/Si-DLC, both the N/Si-DLC and the (Ar + N)/Si-DLC coatings were practically pinhole free. The surfaces of the N/Si-DLC and (Ar + N)/Si-DLC coatings were stain-free and more reflective than the surface of the Ar/Si-DLC coating. The surface reflectivity was observed to increase with increasing proportions of nitrogen in the ion beam. These observations are not yet understood.

3.2 Compositional Analysis. The composition of the Si-DLC films was measured using Rutherford backscattering (2-MeV He^+, 170°) and the simulation program RUMP [8]. In all cases, the relative ratios of carbon (C), Si, and oxygen (O) in the IBAD coatings were found to be approximately the same as the precursor: C:Si:O = 14:1.5:1. This strongly suggests that the siloxane backbone (Si-O-Si-O-Si) of the precursor molecule remains intact during the ion irradiation process, and only C:H and C:C bonds are broken to convert the oil to hard DLC.

The measured compositional differences noted between Ar/Si-DLC, (Ar + N)/Si-DLC, and N/Si-DLC coatings involved the implanted species themselves and their hydrogen (H) content. Si-DLC formed with an argon ion beam contained 3–5 atomic percent argon, and coatings produced using only nitrogen ions contained 7–12 atomic percent nitrogen. In both cases, there is a zone near the surface, approximately equal to the predicted ion range, which is relatively deficient in the implanted species. The coatings produced by nitrogen-containing beams contained nearly twice as much hydrogen (circa 28 atomic percent) as argon-only-produced coatings (12–15 atomic percent).

In the case of Si-DLC produced using a mixture of argon and nitrogen ions, RBS revealed a distinct two-layer structure. In these coatings, a 250-nm layer near the surface contains 0.5 atomic percent argon, but the underlying material contains little or no argon. It is possible that the nitrogen ions, traveling farther into the growing film than the argon ions, displaced the implanted argon either directly or by facilitating its escape through defect production. This effect would be less prominent near the surface, where nitrogen ions interact primarily via electronic stopping, leaving an argon-rich surface layer. The nitrogen content of the coatings appears to be similar to that found using only nitrogen ions (7–12 atomic percent).

3.3 Growth Rate, Microstructure, and Electrical Properties. The Si-DLC coatings produced using N^+, $Ar^+ + N^+$, and Ar^+ alone were 420 nm, 505 nm, and 714 nm thick, corresponding to average growth rates of 7.14 nm/min, 5.05 nm/min, and 4.2 nm/min, respectively (Table 1). The growth rate difference of these coatings may be attributed on the one hand to the higher surface sputtering rate (etching) caused by the heavier argon ion and on the other hand to the higher number of ionization events created by the nitrogen ion, which presumably enhanced the N/Si-DLC growth rate [9]. X-ray

Table 1. Summary of Measured Properties of Si-DLC Coatings Deposited on Silicon With the Assistance of Ar, Ar + N, and N Ion Beams.

Property	Ar	Ar + N	N
Thickness (nm)	420	505	714
Growth Rate (nm/min)	4.2	5.05	7.14
Microhardness (GPa)	10 ± 0.1	14 ± 0.1	11 ± 0.1
Nanohardness (GPa)	10 ± 0.1	12.8 ± 0.8	11.6 ± 1.65
Modulus of Elasticity (GPa)	100 ± 1	160 ± 8	141 ± 14.1
Friction Coefficient μ (average)	0.12	0.12	0.07
Wear Volume (m^3)	1.6×10^{-13}	1.54×10^{-13}	0.86×10^{-13}

analysis showed that all films were amorphous, in agreement with our previous results [6]. The resistivity of all coatings was above 30 kΩcm, which was beyond the maximum measurable value of the four-point probe apparatus used. All coatings appeared to be featureless when examined under an ordinary optical microscope (200X).

3.4 Raman Spectra. The Raman spectra of the Ar/Si-DLC and N/Si-DLC coatings were measured (not shown) and resolved into a "D" peak and a "G" peak associated with the sp^3 and sp^2 bond, respectively. The "D" peak, occurring at 1,340 cm^{-1}, results from defect-induced disorder in the microcrystalline graphite. The "G" peak, occurring at 1,600 cm^{-1}, arises from the scattering sp^2 bonded carbon in graphite crystals. The relative intensity ratio of the two bands is indicative of the disorder present in the material with increased disorder accompanied by increased intensity of the "D" mode. The Si-DLC coatings produced from these two ion beams did not display any significant differences in their Raman spectra, indicating no significant disorder differences between nitrogen- and argon-only samples.

3.5 Fourier Transform Infrared (FTIR) Spectra. Figure 1 shows the FTIR spectra of the Si-DLC coatings. Table 2 shows the peak position and mode assignment for the FTIR Ar/Si-DLC and N/Si-DLC spectra. FTIR spectra were taken for the nitrogen-only and argon-only-produced coatings.

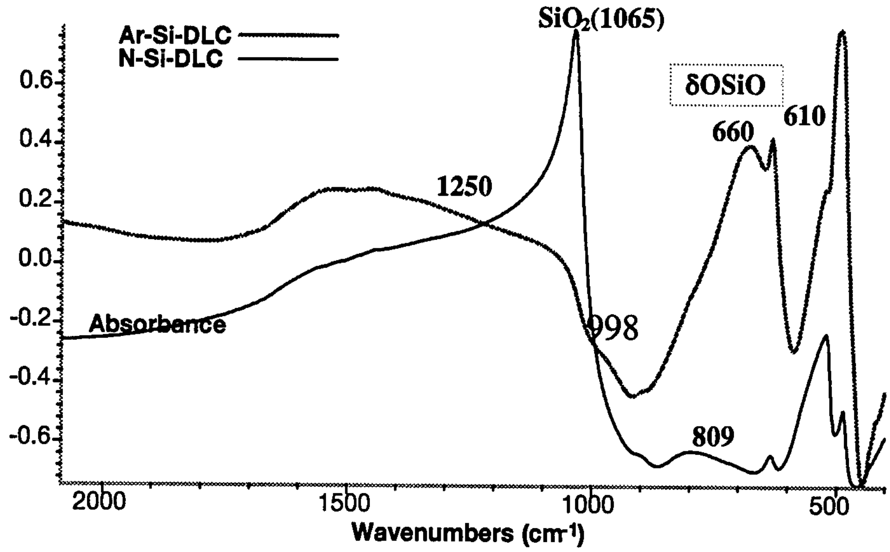

Figure 1. FTIR Spectra of N/Si-DLC and Ar/Si-DLC Coatings on Sapphire.

Table 2. Summary of FTIR Modes

Peak (cm^{-1})	Assignment
610	(δOSiO)[3]
660	(δOSiO)[3]
809	(Si-H)[2]
820	(Si-H)[2]
998	H —C—H H
1,065	(OSiO)[3]
1,250	(Si-CH)[2]

They show 610 and 660-cm^{-1} infrared (IR) modes, which are assigned to Si-O bonds. The 809 and 820-cm^{-1} mode is assigned to the (Si-H)[2] vibrations. Other notable differences between the spectra of the two films are seen for the modes observed at 998; 1,065; and 1,250 cm^{-1}. For the N/Si-DLC coating, there is a sharp mode present at 1,065 cm^{-1} assigned to SiO_2, whereas for the Ar/Si-DLC coating, there is a broad combined mode at 998 and 1,250 cm^{-1}. The mode at 998 cm^{-1} is attributed to $C\text{-}H_3$, while the higher frequency 1,250 cm^{-1} mode is indicative of $Si\text{-}CH_3$ bonds. These results indicate that Si is tetrahedrally bonded to hydrogen for both Ar/Si-DLC and N/Si-DLC coatings. However, SiO_2 bonding is present only in the N/Si-DLC coating.

3.6 Microhardness, Nanohardness, Modulus of Elasticity, Adhesion, and Wear. Knoop microhardness measurements were made using loads of 0.15 N, which correspond to penetration depths of 1,000 nm, larger than the maximum coating thickness for N/Si-DLC of 710 nm. The average Knoop microhardness of the (Ar + N)/Si-DLC coating (uncorrected for substrate effect) was 14 GPa, 27% higher than the Knoop microhardness of the N/Si-DLC (11 GPa) and 40% higher than the Knoop microhardness of the Ar/Si-DLC coating (10 GPa). Nanoindentation measurements were made using a Nano Instruments XP nanoindenter, with a Berkovich three-sided pyramid diamond indenter with controlled penetration depths of 300 nm. The instrument allowed an indenter penetration vs. force curve to be determined allowing the determination of both (nano) hardness and the effective elastic modulus from the slope of the hysteresis curve [7]. These measurements follow the same pattern as the Knoop microhardness ones. The nanohardness of the (Ar + N)/Si-DLC coating was 12.8 ± 0.8 GPa, about 20% higher than the nanohardness of the N/Si-DLC (11.6 ± 1.65 GPa) and 40% higher than the Knoop microhardness of the Ar/Si-DLC coating (10 ± 0.1 GPa). The modulus of elasticity of the (Ar + N)/Si-DLC, N/Si-DLC, and Ar/Si-DLC coatings was 160 ± 8 GPa, 141 ± 14.1 GPa, and 100 ± 1 GPa, respectively.

No delamination was observed while testing the adhesion of the coatings to their underlying silicon substrates either by the so-called Scotch-Tape test or during ball-on-disk wear test measurements. The wear volumes of the N/Si-DLC, (Ar + N)/Si-DLC, and Ar/Si-DLC coatings, determined with the aid of a stylus profilometer, were 0.86×10^{-13} m^3, 1.54×10^{-13} m^3, and 1.6×10^{-13} m^3, respectively (0.54::0.96::1.0). The wear tracks on the surface of all coatings were barely visible to the unaided eye. Extrapolating the model developed by Rao and Lee [9] about the ion implantation on surface properties of polymers on our Si-DLC coatings, we may attribute the improvements in properties to a consequence of cross-linking of the precursor material caused by the ion irradiation. The dual nitrogen-plus-argon irradiation was better because it combined a deeper implant, in the form of nitrogen, along with argon irradiation, which resulted in a shallower but more highly cross-linked layer at the near-surface. Thus, a deeper and graded cross-linked surface region is thought to have been formed. However, the wear rate may also be, at least partially, attributed to the different thicknesses of these Si-DLC coatings. Table 1 shows the microhardness and nanohardness results of all Si-DLC coatings.

3.7 Sliding Friction Coefficient. The variation of the unlubricated friction coefficient of the N/Si-DLC, (Ar + N)/Si-DLC, and Ar/Si-DLC coatings is shown on Figure 2. The unlubricated friction coefficient of the N/Si-DLC coating is, for the most part of the 400-m distance traveled by the steel ball, significantly smaller than the friction coefficient of the (Ar + N)/Si-DLC and Ar/Si-DLC coatings. Meletis, Erdemir, and Fenske [10, 11] have attributed the smaller friction coefficient of their conventional DLC and our Si-DLC coatings to their graphitization due to frictional heating, generated by the rotating steel ball on the coating surface, during the ball-on-disk testing. The beginning of graphitization of the Si-DLC coating is indicated by the abrupt (downward) change of the slope of the curves during the initial 40-m travel distance of Figure 2. However, the ball-on-disk friction coefficient of the N/Si-DLC and (Ar + N)/Si-DLC coatings, unlike the Ar/Si-DLC coating, for reasons not yet understood, increased monotonically during the course of the 400-m traveled distance. The latter may be attributed, as indicated by the FTIR results, to the formation of SiO_2 bonding, observed only in the coatings formed with ion beams containing nitrogen.

4. Conclusions and Future Plans

Synthesis of Si-DLC coatings with N-containing ion beams yields amorphous, nonconductive coatings with lower friction coefficients and significantly lower wear rates than the Si-DLC coatings we have produced to date using only argon ions. This trend is tentatively attributed to the greater ion penetration of the nitrogen ions and to the observed correlation between SiO_2 content and lowered friction. Valance Band X-ray Photoelectron (XPS) and Auger analysis within the wear track is planned to test this correlation.

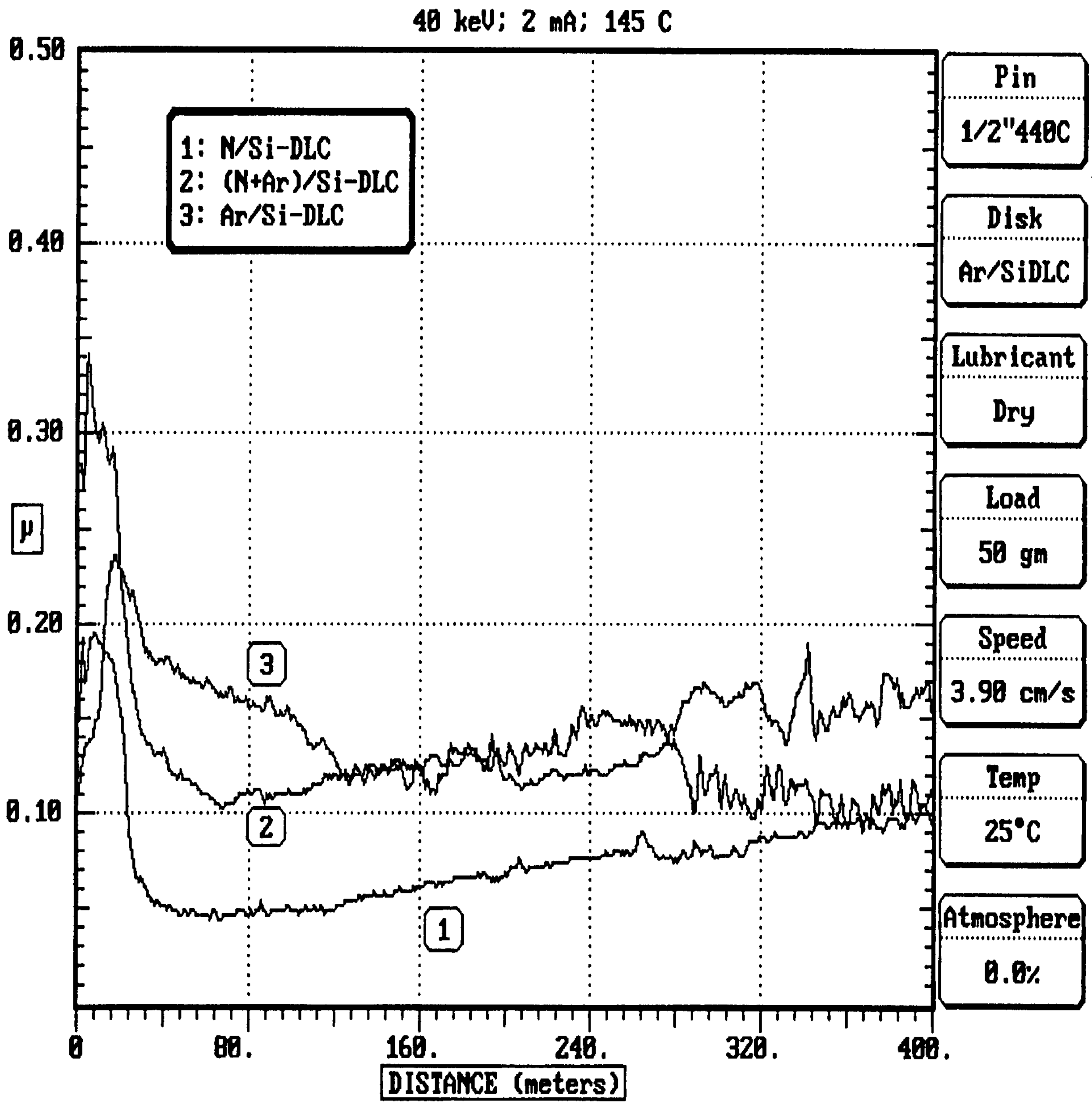

Figure 2. Unlubricated Ball-on-Disk Friction Coefficient of Si-DLC Coatings Synthesized With N (1), (Ar + N) (2), and Ar (3) Ion Beams.

Nitrogen and other light-element ions will be used for the synthesis of the Si-DLC coating on other substrates, including glass and composite materials of practical importance for Army applications. In addition, the effect of the thickness of the Si-DLC coating on its wear and mechanical properties will also be examined.

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