

SYNTHESIS OF SUPERLOW FRICTION CARBON FILMS FROM HYDROGEN-RICH ACETYLENE PLASMAS*

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ABSTRACT

In this paper, we introduce a class of new diamondlike carbon films that can provide superlow friction and wear properties to sliding surfaces in dry nitrogen. The films were produced in a plasma-enhanced chemical-vapor deposition system on AISI M50 and H13 steel substrates by using hydrogen-rich acetylene (C_2H_2) plasmas at room temperature. The friction and wear testing of the films was performed in a pin-on-disk machine under a 10-N load at a velocity of 0.5 m/s. Test results revealed a very close correlation between friction and wear coefficients and the hydrogen content of the gas discharge plasma from which the DLC films were derived. Specifically, the films derived from plasmas with higher hydrogen exhibited increasingly lower friction and wear coefficients than films grown in pure C_2H_2 . The lowest friction coefficient (0.003) was achieved with a film derived from a gas mixture of 90 vol.% H_2 + 10 vol.% C_2H_2 , whereas the friction coefficient of films derived from pure C_2H_2 was 0.35. Similar correlations were observed for wear rates. Specifically, films derived from C_2H_2 were worn out rather quickly and the substrate steel was exposed, whereas films of highly hydrogenated plasmas remained intact and wore at rates 2-3 orders of magnitude lower than those of films obtained from pure C_2H_2 . Based on surface studies and tribological test results, a mechanistic model is presented to elucidate the superlow friction coefficients of films grown in highly hydrogenated plasmas.

Key Words: superlow friction and wear, diamondlike carbon, lubrication mechanism, highly hydrogenated acetylene plasmas.

INTRODUCTION

In recent years, interest in diamondlike carbon (DLC) films has increased tremendously, mainly because, many attractive properties offered by these films can meet the very stringent and increasingly multifunctional application needs of advanced mechanical devices such as gears, bearings, biomedical implants, mechanical seals, computer hard disks, metal- and/or plastic-forming dies, etc.¹⁻⁵. These films are structurally amorphous and can be deposited on a variety of substrates at room temperature. There are several physical and/or chemical-vapor deposition processes such as ion-beam deposition, DC and RF magnetron sputtering, arc physical-vapor deposition, plasma-enhanced chemical vapor deposition (PECVD), and laser ablation that can produce high-quality DLC films on a variety of substrates.⁶⁻¹³ In most of these processes, DLC is derived from a hydrocarbon source gas (e.g., methane, acetylene, ethylene, etc.), but it can also be derived from solid carbon materials such as graphite, glassy carbon, fullerenes, etc. The films derived from hydrocarbons contain hydrogen in their microstructures, and are often referred to as hydrogenated DLC films. In recent years, researchers have developed ways to incorporate several elemental species (e.g., nitrogen, fluorine, boron, silicon, tungsten, titanium, niobium) into DLC films to obtain better optical, electronic, or tribological properties.^{14,15}

Most DLC films are mechanically hard and rigid and hence are able to provide high wear resistance to sliding contact interfaces. Because of their high chemical inertness, they can also

provide low friction and resist chemical or corrosive attack in aggressive environments. Previous studies have shown that the extent of reduction in friction and wear is greatly influenced by several factors. It was found that hydrogen content of the DLC film as well as the ratio of sp^2 to sp^3 bonds can have significant effects on friction and wear. Test environment and ambient temperature were also found to influence friction and wear behavior of these films. With respect to hydrogen content, researchers have shown that hydrogen-free DLC films performed better in moist air than dry air or inert gases, whereas hydrogenated DLCs exhibit superior friction and wear properties in dry and inert test environments.¹⁶⁻²⁰ Several other factors (e.g., counterface material, film thickness, substrate hardness and stiffness, etc.) were also found to influence the friction and wear performance of most DLC films.^{19,21-23} Elevated temperatures were found to have a detrimental effect on the friction and wear behavior of DLC films.²⁴ In particular, hydrogenated DLC films undergo phase transformation at elevated temperatures and became graphitized. The doping of DLC films with certain elements (such as silicon, titanium, tungsten, boron) may retard graphitization and hence enhance the durability of DLC films at elevated temperatures.

Recent systematic studies in our research laboratory have led to the development of a new class of DLC films that afford superlow friction and wear properties to sliding ceramic and steel substrates.^{25,26} These films were produced in a PECVD system that used methane as the source gas. The lowest friction coefficient, 0.001 was measured on a film deposited on a sapphire substrate.²⁶ In this paper, we report on the friction and wear performance of new films derived from pure acetylene (C_2H_2) and $C_2H_2 + H_2$ plasmas. Specifically, we demonstrate that films with superlow friction and wear properties can also be obtained from highly hydrogenated C_2H_2

plasmas. The major emphasis in this paper is placed on the elucidation of the friction and wear mechanisms of the DLC films grown in highly hydrogenated C_2H_2 plasmas.

EXPERIMENTAL PROCEDURES

In this study, we used a PECVD method to produce the DLC films on highly polished surfaces of AISI M50 balls and H13 steel substrates. The surface finish of these steel samples was better than $0.02\ \mu\text{m}$, RMS. The films were 1-1.5 μm thick and were derived from pure C_2H_2 and C_2H_2 + an increasing amounts of hydrogen (up to 90 vol.%). C_2H_2 is an excellent source gas for the deposition of pure and metal-containing DLC films at very high rates. The procedure for forming DLC films on steel substrates by PECVD involved sputter-cleaning of the substrates in an argon plasma for 30 min by applying a 1200-1700 V bias. The substrates were then coated with a 50 – 70-nm-thick silicon bond layer by switching to a sputtering mode and sputtering silicon from a target. In some cases, silane (SiH_4) gas was also used to form the bond layer on the steel substrates. Finally, C_2H_2 or $C_2H_2 + H_2$ gases were introduced into the chamber and the deposition of DLC on the substrates was started. The gas pressure ranged from 10 to 13 mtorr and the RF bias was maintained between 400 and 600 V. The deposition rates in highly-hydrogenated plasmas (e.g., 10% $C_2H_2 + 90\%C_2H_2 + H_2$) was very low, i.e., $0.3\ \mu\text{m}/\text{h}$, while in a C_2H_2 plasma, the rate was more than $3\ \mu\text{m}/\text{h}$. Further details of the deposition process can be found in Refs. 25-27.

The uncoated and DLC-coated samples were tested for friction and wear using a ball-on-disk tribometer in a dry nitrogen environment at room temperature. The contact load was 10 N (which created a peak Hertz pressure of $\approx 1\ \text{GPa}$ between a 9.55-mm-diameter M50 steel ball and steel

disk). The sliding distance and velocity were 5 km and 0.5 m/s, respectively. To measure the friction coefficient of the DLC coating against itself, not against steel, AISI M50 steel balls used in the ball-on-disk experiments were also coated with DLC. The Vickers hardness of the substrates and balls was 8 GPa and their surface roughness was better than 0.05 μm RMS. Each coating was tested twice in dry nitrogen (0% humidity). The test chamber was purged with dry nitrogen for at least 2h after 0% humidity was attained on a hygrometer display unit. Wear volume (W_b) of the steel balls was determined with an optical microscope. Specifically, wear scar diameter and the diameter of the ball were used in the equation $W_b = 3.14d^4/64r$, where r is the ball radius, d is the diameter of the wear scar, and W_b is the wear volume. To simplify the calculations, we assumed that the wear scar was flat. Raman spectra of the films were measured with a Renishaw Raman microscope that uses a HeNe laser at 632.8 nm, with an output power of 25 mW. The focused spot size for the laser beam was 2 μm .

RESULTS

The Raman spectra of the DLC films derived from pure C_2H_2 and $\text{C}_2\text{H}_2 + \text{H}_2$ source gases exhibited broad peaks at $\sim 1494\text{-}1538\text{ cm}^{-1}$ and shouldered peaks at $1320\text{-}1332\text{ cm}^{-1}$. Figure 1 shows the Raman spectrum of a film derived from 100% C_2H_2 and that of a film derived from 10% $\text{C}_2\text{H}_2 + 90\%$ H_2 . The shouldered peaks were somewhat less pronounced on films derived from pure C_2H_2 than films derived from increasingly hydrogenated C_2H_2 plasmas. Overall, the DLC films tested in this study displayed Raman features typical of amorphous DLC films and were consistent with the Raman spectra presented elsewhere.^{28,29} Figure 2 shows a scanning electron microscopy (SEM) cross-sectional photomicrograph of a film derived from a plasma

containing 90% H₂ and 10% C₂H₂. The film seems to have a featureless microstructure with no evidence of a columnar morphology, which is very typical of most PVD hard coatings.

Figure 3 summarizes the friction and wear performance of DLC films derived from various source gases. The friction and wear values in these graphs represent the average of two to three repeat tests. The friction coefficient decreases with increasing hydrogen in the plasma. In general, the higher the hydrogen, the lower the friction coefficient. Figure 4 shows the actual frictional traces of films derived from pure C₂H₂ and 90% H₂ + 10% C₂H₂ plasmas. As is clear, the friction coefficient of the films grown in pure C₂H₂ is the highest (fluctuating between 0.3 and 0.4). Furthermore, the frictional trace of this film is somewhat unsteady. After the sliding tests, we noticed that the film was totally worn out on the ball side of the sliding pair and that the steel substrate was clearly visible. In contrast, the DLC film grown in a gas discharge plasma that consisted of 10% C₂H₂ + 90% H₂ (see Fig. 4) exhibited a smooth friction trace and provided the lowest friction coefficient among all the films tested in this study. When tested under the same conditions, the friction coefficient of an uncoated M50 ball against the H13 steel disk was 0.8.

The wear rates of all of the DLC-coated M50 balls during sliding against DLC-coated H13 disks in dry nitrogen are presented in Fig 3b. Note that these wear rates show a similar trend to that of the friction results presented in Fig. 3a. Specifically, test data show that balls coated in pure C₂H₂ plasma suffer the most wear (i.e., 7.5×10^{-7} mm³/N.m), whereas balls coated with a film in increasingly hydrogenated C₂H₂ plasmas exhibit increasingly lower wear rates. The lowest wear, 2.8×10^{-10} mm³/N.m, was observed on a film that was derived from a plasma that consisted of 10% C₂H₂ + 90% H₂. Figure 5 shows the size, shape, and condition of wear scars formed on two

steel balls. The ball in Fig. 5a was coated with DLC in a pure C₂H₂ plasma and this coating was worn out rather quickly. The ball in Fig. 5b was coated with DLC in 10% C₂H₂ + 90% H₂, and as can be seen, the coating is still intact. The wear rate of the ball in Fig. 5a is 2500 times higher than that of the ball in Fig. 5b. Note that the wear scar formed on the sliding contact spot of the ball in Fig. 5b is rather clean and free of a transfer layer which is often encountered on balls slid against DLC films³⁴⁻³⁷. When tested under the same conditions, the wear rate of an uncoated M50 ball against the uncoated H13 disk was $4.6 \times 10^{-6} \text{ mm}^3/\text{N.m}$.

DISCUSSION

The results presented above suggest that the composition of the source gas plasma used during deposition of DLC films has a dramatic effect on the friction and wear behavior of the films. As is clear from Figs. 3 and 4, the films grown in pure C₂H₂ plasmas wore quickly and exhibited very high friction. The films grown in a highly hydrogenated C₂H₂ plasma (containing 90% H₂) provided the best overall friction and wear performance. The exact mechanism(s) that control friction and wear behavior of DLC-coated surfaces are not yet fully understood, but several mechanisms have been proposed in past years. The generally accepted view is that the low friction of these films is mainly the result of the highly passive nature of their sliding surfaces.³¹⁻
³³ Low friction due to bulk shear is very unlikely, because these films are amorphous, hence have no slip systems. High chemical inertness or passivity of sliding DLC surfaces apparently causes very little adhesion across their sliding contact interfaces and hence lowers friction. Micrographitization of sliding DLC surfaces and the formation of a transfer layer on mating surfaces can also significantly affect friction and wear behavior of DLC films.³⁴⁻³⁷ Furthermore,

test parameters (such as ambient temperature, velocity, relative humidity, contact pressure, environment, etc.) can affect sliding friction and wear behavior of DLC films.^{16,19,20}

It is well known that rubbing one rough surface against another generates large amounts of traction, and hence high friction, whereas rubbing one smooth surface against another often produces only low friction; this is particularly true for diamond and DLC films which provide low friction through an interface slip mechanism.^{2,38,39} In addition to testing parameters and physical roughness, the extent of chemical or adhesive interactions across a sliding interface can play a major role in friction. These bonds may result from a wide variety of short- and long-range chemical/physical interactions such as covalent, ionic, metallic, van der Waals, electrostatic, capillary forces, etc. Covalent bonding can occur between sliding diamond surfaces in ultrahigh vacuum and causes very high friction (i.e., friction coefficients >1).^{38,40} However, when moisture or other chemically active gaseous species (such as oxygen, hydrogen, fluorine, etc.) are introduced into the test chamber, the friction coefficient of diamond drops precipitously to 0.05 or less. This is thought to be the result of chemical passivation of the free σ bonds on the surface and thus the elimination of strong covalent interactions across the sliding interface. This explanation is the most widely accepted for the self-lubrication mechanism of diamonds^{30-33,40} High friction (0.8) between uncoated M50 steel and uncoated H13 tested in this study may have been largely due to the formation of strong metallic bonds across their sliding interface. Briefly, the making and breaking of bonds at the sliding interface largely controls the extent of friction between sliding DLC surfaces.

Besides very strong covalent bonding, some other forces (van der Waals, electrostatic, capillary, π - π^* interactions) may also be present at the sliding contact interfaces of diamond or DLC films. The van der Waals attraction and π bonding are more relevant to graphite than to diamond or high-quality DLCs. When the sliding surfaces of diamond or DLC are graphitized under the influence of high thermal or mechanical loadings at high sliding velocities and/or temperatures, the extent of π -bonds may become significant and can dominate the frictional performance of these materials.^{2,34-37,41} Furthermore, certain DLC films may contain high proportions of sp^2 -bonded or graphitic carbon precursors in their microstructures; hence the residual π bonding in these films may also be substantial. Finally, in open air, several mono-layers of water molecules and/or other gaseous species may have been present on the sliding surfaces of DLC films. These adsorbed layers may easily form a meniscus in and around the real contact spots that can in turn create residual capillary forces. In vacuum or well-controlled inert-gas environments, such forces are greatly reduced or are essentially absent.

In this study, the most striking observation was the existence of a close correlation between the source gas composition and the friction and wear performance of the resultant DLC films. Specifically, the DLC films produced in source gases with increasing amounts of hydrogen exhibited lower and lower friction and wear coefficients (see Figs. 3 and 4), with the lowest friction coefficient, 0.003, provided by a DLC film grown in a 10% C_2H_2 + 90% H_2 plasma. The films grown in pure C_2H_2 plasmas exhibited the highest friction and wear. These observations suggest that the amount of hydrogen in the gas discharge plasma plays a critical role in the friction and wear performance of the resultant DLC films. For the superlow-friction coefficients of DLC films produced in our study, we provide the following explanation.

As discussed earlier, hydrogen can chemically bond to and effectively passivate the free σ -bonds of carbon atoms in diamond and related materials and make these materials chemically very inert.³⁰⁻³³ It is important to remember that the C-H bonding is extremely strong (stronger than single C-C, C-O, or C-N bonds). We believe that when extra hydrogen is used during DLC deposition, several important events and control the structural chemistry of the resultant DLC films that ultimately govern the friction and wear performance. First, it is logical to claim that the amount of hydrogen in the bulk, as well as on the surface, of the DLC films will increase substantially. Most of the hydrogen atoms are expected to be paired with the σ bonds of carbon atoms, but some unbonded free hydrogen may also be present as interstitial species in the bulk. Increased hydrogen density in the bulk and on the surface should effectively diminish or even eliminate the possibility of any unoccupied σ bonds remaining and participating in adhesive interactions during sliding contact.

Second, hydrogen is known to effectively etch out or remove the sp^2 -bonded or graphitic carbon precursor from the film surface and thus prevent the formation of planar graphitic phases and/or cross linking that can give rise to π bonding.⁴² When DLC films are prepared in a highly hydrogenated plasma and under energetic hydrogen ion bombardment, strong C-H bonding rather than C=C double bonding should be favored. As explained above, the existence of residual π bonding that can be the result of C=C double bonds in DLC can give rise to high friction. Such a C=C double bonding may have been present in films derived from pure C_2H_2 .

Finally, we believe that perhaps some of the carbon atoms (at least the ones on the near surface) were dihydrated, that is that two hydrogen atoms were bonded to each carbon atom on the surface. This can occur on the unreconstructed (100) surfaces of diamond structures under some special or supercritical conditions⁴² that may have been created by the energetic hydrogen bombardment in a highly hydrogenated gas discharge plasma. The existence of dihydrated carbon atoms on the surfaces will increase the hydrogen density of these surfaces and provide better shielding or polarization of strong σ bonds and thus superlow friction.

As mentioned above, other influences such as van der Waals and capillary forces, as well as electrostatic attraction, can also be present and contribute to the friction between two sliding surfaces. In fact, when we repeated some of the tests in open air, the friction coefficients of films grown in 10% C_2H_2 + 90% H_2 increased to values as high as 0.06 (depending on the relative humidity). Because we ran the friction tests in a clean, dry nitrogen environment, the extent of capillary forces due to moisture precipitation on or around the real contact spots should be minimal or essentially absent. As for the van der Waals forces, they will always be present at the sliding contact interfaces but their relative contributions to the overall frictional force should be rather insignificant, because we used very high contact loads in our experiments. As for the possibility of electrostatic attraction, we feel that because the DLC films are in generally dielectric, their sliding surfaces can certainly accumulate static electrical charges. The main question is whether these charges will cause attraction or repulsion. When the free electrons of hydrogen atoms pair with the dangling σ bonds of carbon atoms, the electrical charge density is permanently shifted to the other side of the nucleus of the hydrogen atom and away from the surface. Such a shift in charge density allows the positively charged hydrogen proton in its

nucleus to be closer to the surface than the electron that is used up by the σ bond of the surface carbon atoms. We believe that the creation of such a dipole configuration at the sliding interface should give rise to electrostatic repulsion rather than attraction between the hydrogen-terminated sliding surfaces of the DLC films.

Briefly, we believe that hydrogen plays an essential role in both the type and extent of chemical/physical interactions and hence in the friction of DLC films. Films grown in hydrogen-rich C_2H_2 plasmas are more likely to contain more hydrogen on their sliding surfaces and in their microstructures than the films grown in pure C_2H_2 so these films are chemically more inert, and their covalent σ bonds (which can cause adhesion/friction) are absent.

CONCLUSIONS

Based on the results of this experimental study, the following conclusions can be drawn.

1. DLC films grown in pure C_2H_2 plasmas exhibit very poor friction and wear properties when tested in a dry nitrogen environment.
2. DLC films grown in highly hydrogenated C_2H_2 plasmas provide superlow friction and wear properties.
3. The large differences in friction and wear of these DLC films can be attributed to the difference in hydrogen concentration of the source gases from which they were derived.
4. Mechanistically, overhydrogenation of DLC films provides a higher degree of surface passivation, which is essential for reduced adhesion and hence friction.

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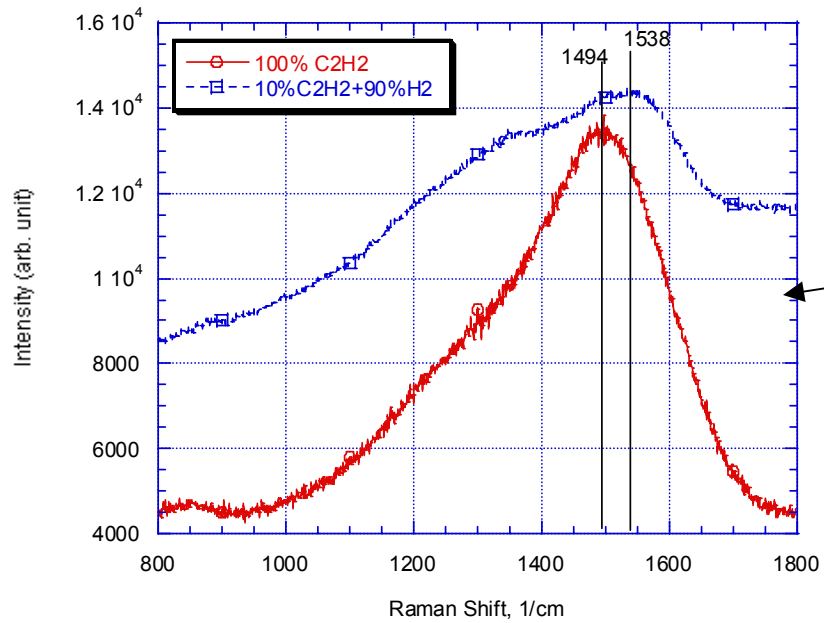


Figure 1. Raman spectra of DLC films derived from pure C_2H_2 and 10% $C_2H_2 + 90\% H_2$

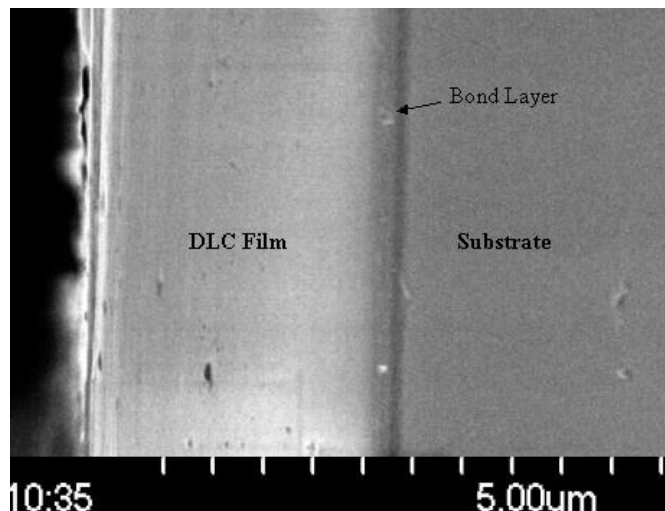
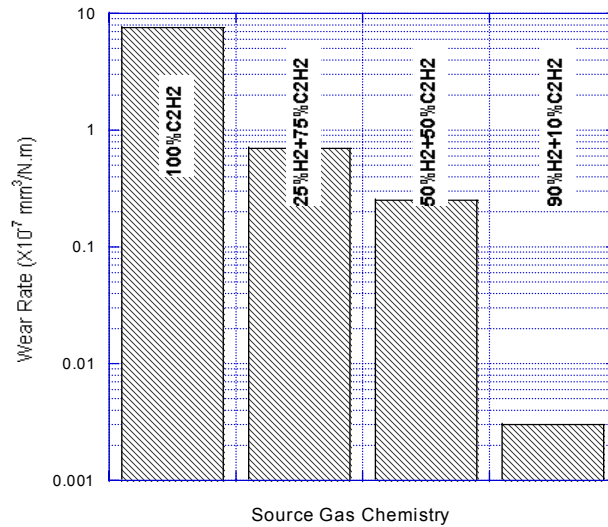
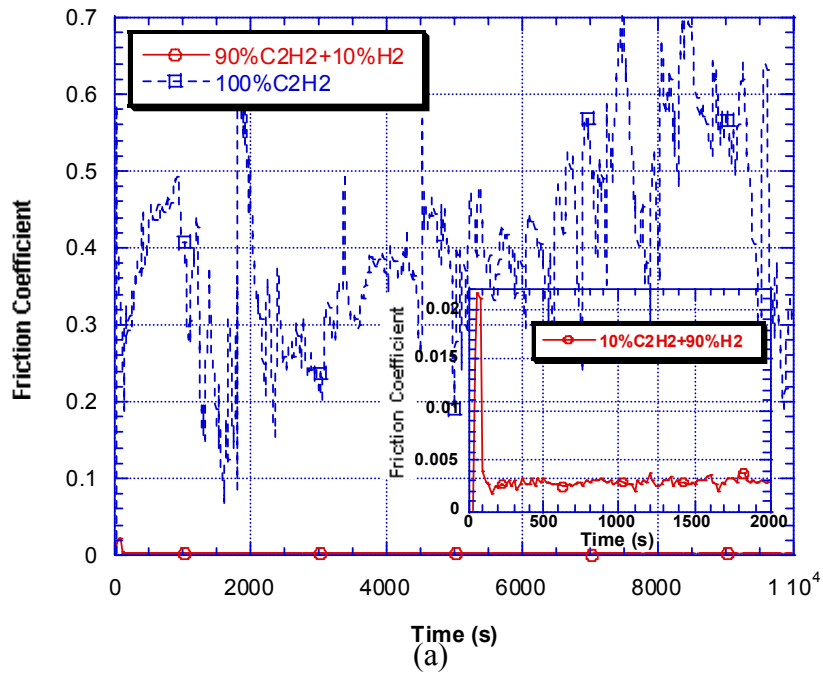


Figure 2. Cross-sectional SEM photomicrograph of DLC film grown in 90% $H_2 + 10\% C_2H_2$.



(b)

Figure 3. (a) Friction and (b) wear performance of DLC films produced in various source gases

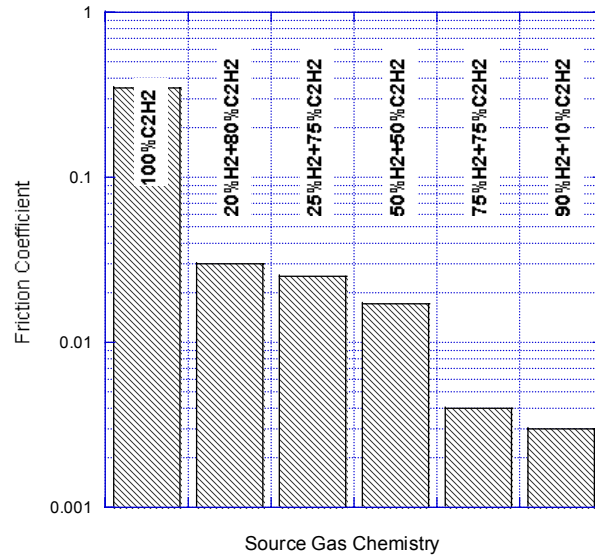
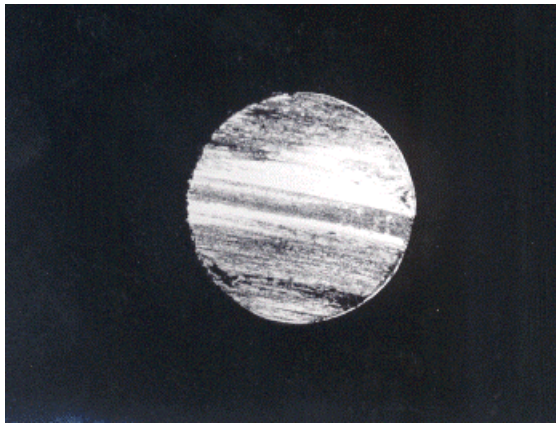
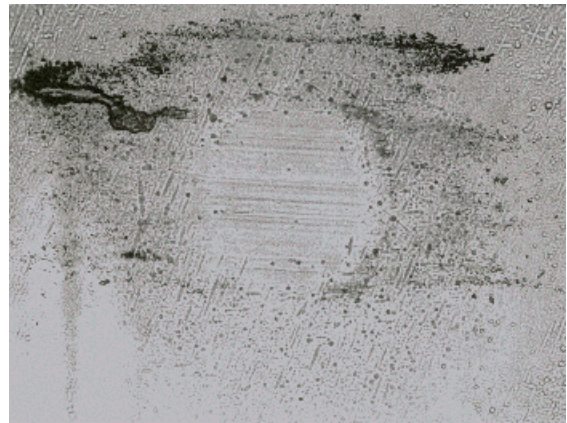


Figure 4. Typical friction coefficient traces of DLC films derived from pure C₂H₂ and 10% C₂H₂ + 90% H₂



(a) (Mag. 37X)



(b) (Mag. 155X)

Figure 5. Photomicrographs of wear scars formed on steel balls coated with DLC in (a) pure C₂H₂ and (b) 90% H₂ + 10% C₂H₂