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Title: OPTICAL AND TRIBOLOGICAL PROPERTIES OF
DIAMOND-LIKE CARBON FILMS SYNTHESIZED BY
PLASMA IMMERSION ION PROCESSING

Author(s): Xiao-Ming He, MST-8
Kevin C. Walter, MST-8
Jean-Francois Bardeau, CST-1
Michael Nastasi, MST-8
S-T Lee, City University of Hong Kong
X.S. Sun, City University of Hong Kong

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Optical and Tribological Properties of Diamond-like Carbon Films Synthesized by Plasma Immersion Ion Processing

X.M. He, K.C. Walter, J. F. Bardeau, and M. Nastasi

Los Alamos National Laboratory, Materials Science and Technology Division

Los Alamos, New Mexico 87545, USA

S.-T. Lee and X.S. Sun

Department of Physics and Materials Science, City University of Hong Kong, Hong Kong

ABSTRACT: Hard diamond-like carbon (DLC) films have been prepared on PMMA (polymethyl methacrylate), glass, and Si(100) substrates using C₂H₂-Ar plasma immersion ion processing (PIIP). The composition, structure, and properties of the films were investigated with regard to variation of the deposition parameters. It was found that the modulation of reactive gas composition during PIIP could enhance the formation of DLC films with an increased sp^3 bonding structure, improved surface smoothness, high density and high hardness. An optimal combination of good optical properties and high hardness was highly dependent on the control of hydrogen content in the DLC films. Tribological tests showed that DLC-coated glass and PMMA samples exhibited a reduced friction coefficient and enhanced wear resistance relative to uncoated glass and PMMA materials. The effects of ion energy and gas composition during PIIP deposition on the formation of optically transparent and wear resistant DLC films are discussed.

I. INTRODUCTION

The study of amorphous diamond-like carbon (DLC) films has been a very active area of condensed matter physics in recent years, as the films exhibit unique properties similar to those of diamond materials [1-3]. Being chemically inert and mechanically hard, DLC films can be used as a protective coating against corrosion and wear [1,4]. The functional properties of DLC films, such as low conductivity, optical transparency, and sensitive photo-induced conductivity, enable the films to be applied as coatings for an insulator, wide band-gap semiconductors, or optical devices used in electronic and optical areas [2,5,6]. DLC films can be deposited at room temperatures at high deposition rates using plasma-based deposition methods [1-3]. Recently, plasma immersion ion processing (PIIP) techniques, which combined the benefits of ion beam assisted deposition and plasma source ion implantation, have been shown to provide a nonlinear-of-sight deposition of DLC films on room temperature and large-scale substrates [7,8]. Investigation has shown that the composition and properties of DLC films can be functionally changed by the modulation of the negative bias voltage applied to substrates during PIIP [9,10]. Hard and cohesive DLC films more than 5 μm thick have been prepared on complicated-shaped mechanical parts to enhance their surface properties.

The present work will study the composition, structure, and properties of the DLC films with respect to changes of the reactive gas-flow ratio of C_2H_2 to Ar ($F_{\text{C}_2\text{H}_2}/F_{\text{Ar}}$) during PIIP deposition. The investigation of the optical and mechanical properties were comparatively performed on the DLC films deposited on different substrates such as PMMA (polymethyl methacrylate) plastics, glass and Si(100). The systematical analyses of the obtained results show the parameters needed for the optimal deposition of optically transparent and wear resistant DLC films by using PIIP techniques.

II. EXPERIMENTAL PROCEDURES

The preparation of the substrate materials and the PIIP deposition equipment are the same as those reported in our previous work [9,10]. Prior to film deposition, the substrates were subjected to Ar^+ sputter cleaning using a -600 V pulsed bias and an incident dose of $\sim 5 \times 10^{16}$ cm^{-2} to remove surface contaminants or oxides. The transition from surface sputtering to DLC deposition was accomplished by slowly adding C_2H_2 to the existing inductive Ar plasma to promote better adhesion of the DLC film to the substrates. Film preparations were carried out at a constant rf power of 400 W and a negative dc pulsed-bias voltage of -150 V. The pulse width was 20 μs and the pulse frequency was 10 kHz. Under these conditions, the DLC films were synthesized by changing the reactive gas-flow ratios $F_{\text{C}_2\text{H}_2}/F_{\text{Ar}}$ from 0.17 to 0.75 at a fixed gas pressure of 0.13 Pa. Other details for film deposition, such as the base vacuum and the substrate temperature during the deposition, have also been reported elsewhere [10].

The DLC films were prepared on different substrate materials with a thickness of about 220 nm. The film thickness was measured using a Dektak IIA Surface Profilometer. The surface morphology of DLC films was characterized using an extended Multimode Nanoscope IIIA atomic force microscope (AFM). The composition and density of the DLC films were measured and calculated using the rutherford backscattering spectrometry (RBS) and an elastic recoil detection (ERD) technique. The RBS and ERD spectra were measured under the same conditions in ref. [10] and analyzed using RUMP simulation [11]. Raman measurements on DLC films were performed at room temperature with a Renishaw Raman System 2000 imaging microscope using a 5 mW HeNe (514.5 nm) laser. The hardness of the DLC films was measured using an indentation depth of ~ 50 nm with a Nano-indenter[®] II operated in continuous stiffness mode in which fused silica was used as a control material for verifying tip calibration.

An ellipsometer (Rudolph Model AutoEIII, Rudolph Research) operating at 632.8 nm and at 70° angle of incidence was used to measure the refractive index and the extinction coefficient of the DLC films on Si (100). The methods for measurement and calculation of these data are the same as those reported in ref. [10]. The visible light transmittance or absorbance of the DLC films formed on PMMA and glass was measured by an ultraviolet/visible (UV/VIS) spectrometer (Varian Cary-1C) to evaluate the transparency of the DLC films in the visible light range of 350-770 nm. The absorption coefficients of DLC films were calculated based on the obtained absorbance and thickness data. The optical band gap was then determined by fitting the absorption data to a Tauc relation [2].

The wear tests, to explore tribological properties of the DLC films deposited on different materials, were carried out using a pin-on-disk tribometer at room temperature and in air with ~15 % relative humidity (RH). A smooth, 6 mm diameter ruby ($\text{Al}_2\text{O}_3\text{:Cr}$) ball served as the pin for the tests of DLC-coated Si (100) and glass samples, and a HDPE (high density polyethylene) ball, the same diameter as ruby ball, was used as the pin for DLC-coated PMMA samples. The wear track was 3 mm in diameter; a sliding speed of 1.9 cm/s (120 rpm) was employed. The DLC-coated Si(100) and glass samples were wear tested using a maximum of 15,800 wear cycles at an applied load of 4.5 N. The DLC-coated PMMA samples were tested with the same wear cycles but at a wear load of 1.5 N. As the hardness and elastic properties of HDPE are similar to those of PMMA, the uses of a HDPE ball as the pin at the load of 1.5 N do not deformed the PMMA in the wear track, and thus prevent the delamination of DLC films from PMMA surface. The coefficient of friction was electronically monitored and recorded during the tests. After the wear test, the cross sectional area of the wear tracks, the volume of film removed and the wear coefficient (K) were measured and calculated using the approach described in ref. [12].

III. RESULTS

Figure 1(a) shows the hydrogen content and density of the DLC films prepared on Si(100) wafers at various $F_{C_2H_2}/F_{Ar}$ ratios. It is clear that by increasing $F_{C_2H_2}/F_{Ar}$ from 0.17 to 0.75, the hydrogen content in the formed DLC films is increased from 24.5 to 33.1 at. %, while the film density is reduced from 2.78 to 1.83 g cm⁻³. The influence of the variation of the $F_{C_2H_2}/F_{Ar}$ ratio on the hardness and film growth rate is shown in Fig. 1(b). The experimental observation indicated that the growth rate of DLC films on both PMMA and glass was quite similar and almost one order of magnitude higher than that of DLC films on Si(100) wafers. The increase of the $F_{C_2H_2}/F_{Ar}$ ratio results in the increase of the deposition rate and the decrease of the hardness of DLC films, as the data shown in Fig. 1(b). Especially, the hardness of DLC films is reduced from 31 to 17 GPa when the $F_{C_2H_2}/F_{Ar}$ ratio is increased from 0.17 to 0.75. Summarily analyzing the data in Fig. 1, it can be found that the increase of hydrogen contents in DLC films is closely correlated with the reduction of both hardness and mass density. However, when the PIIP process is carried out at the $F_{C_2H_2}/F_{Ar}$ ratio less than 0.5, the DLC films can be deposited with a relative stable growth rate of about 0.025 nm s⁻¹ on Si(100) and 0.042 nm s⁻¹ on both PMMA and glass materials. The resultant DLC films on Si(100) have a lower hydrogen content of less than 30 at %, higher density of above 2.2 g cm⁻², and higher hardness of around 30 GPa.

The Raman spectra of the DLC films prepared at different $F_{C_2H_2}/F_{Ar}$ ratios are shown in Fig. 2. The in-side plot is a typical example of Raman spectra decomposed into bands with Gaussian line shapes, after subtracting a linear background due to luminescence. The fitted line agrees well with the experimental data points. It can be seen that Raman spectra of DLC films all consist of a relatively sharp peak at around 1550 cm⁻¹ and a shoulder at around 1345 cm⁻¹, named as G band and D band respectively. The detail analyses of Raman spectra will be reported

elsewhere [13]. It was found that the decrease of $F_{C_2H_2}/F_{Ar}$ ratios from 0.75 to 0.17 resulted in the shift of both G and D peak positions to lower Raman frequency, and the increase of the G-peak width [full width at half maximum (FWHM)][13]. According to the analyses of McCulloch *et al.* [14] and Tamor and Vasell [15], the changes in Raman spectra indicate that the reduction of $F_{C_2H_2}/F_{Ar}$ ratios enhances the formation of DLC films with more fourfold coordinate bonds, high density and high hardness that are confirmed by the results shown in Fig.1.

The refractive index and extinction coefficient of the DLC films formed on Si(100), as shown in Fig. 3, are both decreased with the increase of $F_{C_2H_2}/F_{Ar}$ ratios. Notably, the refractive index decreases from 2.2 at a $F_{C_2H_2}/F_{Ar}$ ratio of 0.17 to 1.82 at a $F_{C_2H_2}/F_{Ar}$ ratio of 0.75, while the extinction coefficient declines from 0.3 to 0.14. As the reduction of $F_{C_2H_2}/F_{Ar}$ ratios also decrease the hydrogen content and increase the density and hardness of DLC films, it can be seen from the data in Figs.1 and 3 that the large refractive index is correlated with the low hydrogen content, high density and high hardness. This observation is in agreement with previous work, where it was observed that the refractive index scaled with hardness and density of DLC films [2,16]. However, the increase of both the refractive index and extinction coefficient suggests the reduction of the optical transmittance of the formed films.

Figure 4(a) shows the absorption coefficients of DLC-coated PMMA samples, prepared at different $F_{C_2H_2}/F_{Ar}$ ratios, as a function of the photon energy in the visible wavelength range. The increases of the absorption coefficients are correlated with the decrease of the $F_{C_2H_2}/F_{Ar}$ ratio in PIIP depositions, as deduced from the results shown in Fig. 3. It was found that at the same deposition conditions, the variation of $F_{C_2H_2}/F_{Ar}$ ratios exhibited a similar tendency on the influence of the absorption coefficient of DLC films deposited on the glass.

The optical band gap, obtained by the extrapolation of the linear portion of the Tauc curves towards the abscissa [2], as a function of the $F_{C_2H_2}/F_{Ar}$ ratio is shown in Fig. 4(b). It is apparent that the values of the optical gap energies of DLC films on PMMA and glass are quite similar. This implies that the films possess stable optical properties despite their formation on different substrates. In addition, the energy of the band gap increases with increasing the $F_{C_2H_2}/F_{Ar}$ ratio and reaches a maximum value of ~ 2.15 eV when the DLC film is formed at a $F_{C_2H_2}/F_{Ar}$ ratio of 0.75. Associating the data in Fig.4 with those in Figs. 1 and 3, it can be seen that the density, hardness, refractive index, and optical gap energy can all be changed with respect to the hydrogen content that is determined by the variation of the $F_{C_2H_2}/F_{Ar}$ ratio during film growths. The analyses suggest that to prepare hard, dense and transparent DLC films, the hydrogen content should be controlled to less than 30 at. % in the DLC films. This can be approached by modulations of the $F_{C_2H_2}/F_{Ar}$ ratio in a range of 0.25-0.5 during PIIP process. The resultant films do not only exhibit high density and high hardness, but also the good optical properties such as lower absorption coefficients and higher optical gap energies of 1.8-1.9 eV (see Fig.4).

For the tribological investigation, the DLC film prepared at a $F_{C_2H_2}/F_{Ar}$ ratio of 0.33 was selected to perform the wear tests. The friction coefficients of the DLC films formed on Si (100), glass, and PMMA as a function of the wear cycle are shown in Fig. 5. It is clear that DLC-coated Si (100) samples show the lowest friction coefficients of ~ 0.03 . In comparison, DLC coated glass and PMMA samples exhibit higher friction coefficients of 0.22-0.24. However, the data in Fig.5 show that the friction coefficient of DLC-coated glass and PMMA samples are reduce by 50-65 %, as compared with the friction coefficients of these bare materials. Figure 6(a), (b) and (c) show the three-dimensional AFM morphology of the DLC films on Si(100), glass, and PMMA substrates respectively. It is clear that the DLC-coated Si(100) sample exhibits a smoother surface than those of the DLC-coated glass and PMMA samples. AFM measurements indicated that the original

surface root-mean-square (RMS) roughnesses of glass and PMMA, about 1.0 and 8.5 nm respectively, were much higher than that of Si(100) wafers, about 0.32 nm. It was found that after DLC deposition, the RMS roughnesses of DLC-coated Si, glass, and PMMA were all reduced to 0.06, 0.56, and 4.7 nm, respectively. However, the RMS roughnesses of DLC-coated glass and PMMA are still much higher than that of DLC-coated Si(100). Previous work has shown that the friction coefficient of DLC films is correlated with surface roughness during the sliding test [12].

Table I shows the RMS roughness, hardness and tribological properties of DLC films deposited on Si(100), glass and PMMA substrates. The DLC films deposited on glass and PMMA have lower hardnesses and higher RMS roughness as compared with those of DLC-coated Si (100) samples. As PMMA is very soft and the thickness of DLC films is only ~220 nm, the measured hardness of DLC on PMMA is actually a composite value of DLC films and PMMA substrate [17]. However, the hardness of DLC-coated glass and PMMA samples is still one and two times higher than that of glass and PMMA respectively. A possible explanation on the differences of the deposition rate, hardness and RMS roughness among these materials postulates the different insulating properties of these materials that affect the substrate bias and ultimately the ion bombardment during film growth. The data in Table I show that the differences in both surface roughness and hardness seriously affect the tribological performances of DLC films on different substrates. When a wear test is carried out at a load of 4.5 N the DLC-coated Si(100) sample exhibits the lowest wear coefficients of $6.1 \times 10^{-11} \text{ mm}^3/\text{Nm}$. Comparatively, DLC-coated glass samples show a wear coefficient of $50.9 \times 10^{-10} \text{ mm}^3/\text{Nm}$, two orders of magnitude higher than those of DLC-coated Si(100) samples. However, at the load of 4.5 N, the wear volume and wear coefficient of the DLC-coated glass are much lower than the values of bare glass, as seen from table I, although the glass sample was worn only 1800 wear cycles at a load of 1.5 N. Similarly, when the wear test is carried out at a load of 1.5 N, both the wear volume and wear coefficient of DLC-coated

PMMA samples are at least two orders of magnitude lower than those of bare PMMA materials. The results in Table I confirm the strong enhancement of hardness and wear resistance on glass and PMMA materials after deposition of the DLC films.

IV. DISCUSSION

For the deposition of DLC films using PIIP process, changes of only gas composition or the $F_{C_2H_2}/F_{Ar}$ ratio will affect the ratio of the ion densities such as Ar^+ to $C_2H_2^+$ in the plasma, and in turn the ion bombardment on the growing films. The low energy ion bombardment, induced by the negative bias voltage, can promote the formation of sp^3 bonds and the removal of hydrogen by sputtering during the film growth, which leads to the formation of hard a-C:H DLC films [1,3]. In the C_2H_2 -Ar PIIP operations, a low-bias voltage of -150 V has been shown to provide an optimal low-energy condition for ion bombardment on the deposition of DLC films with an enhanced fraction of sp^3 -carbon bonding and high hardness [9]. At a constant gas pressure of 0.13 Pa, increased $F_{C_2H_2}/F_{Ar}$ implies a reduction in the Ar^+ ion density in the plasma, which lowers and broadens the impinging ion energy distribution and weakens the Ar^+ ion bombardment. Therefore, the reduction of the $F_{C_2H_2}/F_{Ar}$ ratio enables more Ar^+ ion produced with energy close to that induced by the bias voltage, to bombard the growing films for assisting depositions. The results on Raman spectra, ion-beam analysis and nano-indentation used in this study have shown that the DLC films formed at a $F_{C_2H_2}/F_{Ar}$ ratio less than 0.5 exhibit increased sp^3 bond density, high density and high hardness.

It is well known that the amount of hydrogen in the film can stabilize the sp^3 bonding to form an a-C:H DLC microstructure [18]. In addition, the presence of hydrogen has been shown to enhance the formation of a transfer layer, which has a lubricating effects to keep the films with

low-friction coefficients in the wear tests [4]. However, the hydrogen content, scaled with the density of C-H bonds in the film, substantially affect the properties of DLC films, such as the optical gap, density, hardness, *etc* [1,3]. In this study, reduction of the $F_{C_2H_2}/F_{Ar}$ ratio enhances the ion impingement on growing films and reduces the hydrogen contained in the films, leading to the formation of DLC films with high sp^3 -bonded fraction, high hardness and high density. In contrast, the DLC films synthesized at a $F_{C_2H_2}/F_{Ar}$ ratio of 0.75 shown a higher hydrogen content of 33 at % and a higher optical band gap of 2.1 eV, but a lower hardness of 17 GPa (see Figs. 1 and 4). The optimum combination of the optical and mechanical properties with respect to the hydrogen content can be reached by improving the ion bombarding conditions through modulating the PIIP parameters. The present study confirms that at a low bias of -150 V and a low pressure of 0.13 Pa the reduction of the $F_{C_2H_2}/F_{Ar}$ ratio to 0.25-0.5 promised the synthesis of the DLC films with a lower hydrogen content of 25-29 at. %, higher hardness of 26-30 GPa, and high density of 2.1-2.65 g cm⁻³, respectively. The resultant films also exhibit good optical properties such as refractive indices of 1.9-2.0 and optical band gaps of 1.8-1.9 eV.

Mechanical hardness and wear resistances are the properties desired for the optically transparent DLC used as a surface coating on optical devices. Our comparative investigation indicates that hard DLC films can be deposited on Si (100), glass and PMMA substrates by using PIIP techniques. The values of the optical gap energies are nearly identical for the DLC films deposited on glass and PMMA. Although the DLC-coated glass and PMMA samples exhibit the different deposition rate, hardness and RMS roughness with those of Si-coated samples, deposition of the DLC film notably smoothed the surface and increased the surface hardness of these materials. The results of the wear tests showed large reductions in the friction coefficient and wear coefficient of glass and PMMA after deposition of DLC coatings. Since PIIP is a scalable process and is not restricted to line-of-sight processing geometries, the results in this

study suggest that the deposition of DLC films by PIIP is promising for applications of hard, wear-proof, and optically transparent coatings on optical materials.

V. CONCLUSION

Optically transparent and wear-resistant DLC films have been prepared on Si(100), glass, and PMMA materials using a C₂H₂-Ar PIIP method. Under constant conditions of a negative bias of -150 V, a rf power of 400 W and a low gas pressure of 0.13 Pa, the reduction of the F_{C₂H₂}/F_{Ar} ratio less than 0.5 was shown to provide the optimum energy conditions for ion impingement on growing films. The formed DLC films had enhanced sp³-bonding structure, high hardness of ~30 GPa, high density of 2.2-2.65 g cm⁻³, and improved surface smoothness. The optimal combination of high hardness above 26 GPa, reference indices of 1.9-2.0, and optical band gap of ~1.85 eV was reached by modulating the preparing parameters to control the hydrogen content (to less than 30 at %) in the DLC films. Deposition of PIIP DLC films on glass and PMMA reduced the surface roughness and increased the surface hardness of these materials, which leads to the reduced friction coefficient and enhanced wear resistance. Notably, the wear volume and wear coefficients of DLC-coated PMMA are at least tow orders of magnitude lower than those of uncoated PMMA. These results show that the PIIP technique is capable of producing functional DLC coatings on optical devices.

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Table I. The RMS and properties of DLC films on different substrates.

Materials	RMS (nm)	Hardness (GPa)	Wear Load (N)	Wear volume (10^{-6} , mm ³)	K (10^{-10} , mm ³ /Nm)
Glass*	1.0	8	1.5	7800.0	74400
PMMA	8.5	0.31	1.5	60970.0	40500
DLC/PMMA	4.7	0.96**	1.5	660	414
DLC/Glass	0.56	17	4.5	287	50.9
DLC/Si(100)	0.06	30	4.5	60	0.61

*: The wear cycles on the glass substrate is only 1800 turns.

** : Hardness measurements reflect a composite value of DLC films and PMMA [17].

LIST OF FIGURE CAPTIONS

Fig. 1 (a) Hydrogen content and density; and (b) hardness and deposition rate of DLC films prepared by PIIP at different $F_{C_2H_2}/F_{Ar}$ ratios. The film thickness is ~ 220 nm.

Fig. 2 Raman spectra of DLC films prepared by PIIP at different $F_{C_2H_2}/F_{Ar}$ ratios. The film thickness is ~ 220 nm. The inner plot is an example of Raman shift decomposed into the G and D bands with Gaussian line shapes, after subtracting a linear background due to luminescence.

Fig. 3 Refractive index and extinction coefficient of DLC films prepared at different $F_{C_2H_2}/F_{Ar}$ ratios. The film thickness is ~ 220 nm.

Fig. 4 (a) Absorption coefficients and (b) optical band gap of DLC films synthesized by PIIP at different $F_{C_2H_2}/F_{Ar}$ ratios. The film thickness is ~ 220 nm.

Fig. 5 The friction coefficient (μ) vs sliding cycles for the DLC films prepared on different substrates at a gas pressure of 0.13 Pa and $F_{C_2H_2}/F_{Ar} = 0.33$. The film thickness is ~ 220 nm.

Fig. 6 AFM morphology of the DLC films prepared on (a) Si(100), (b) glass, and (c) PMMA at a gas pressure of 0.13 Pa and $F_{C_2H_2}/F_{Ar} = 0.33$. The film thickness is ~ 220 nm.

Figure 1

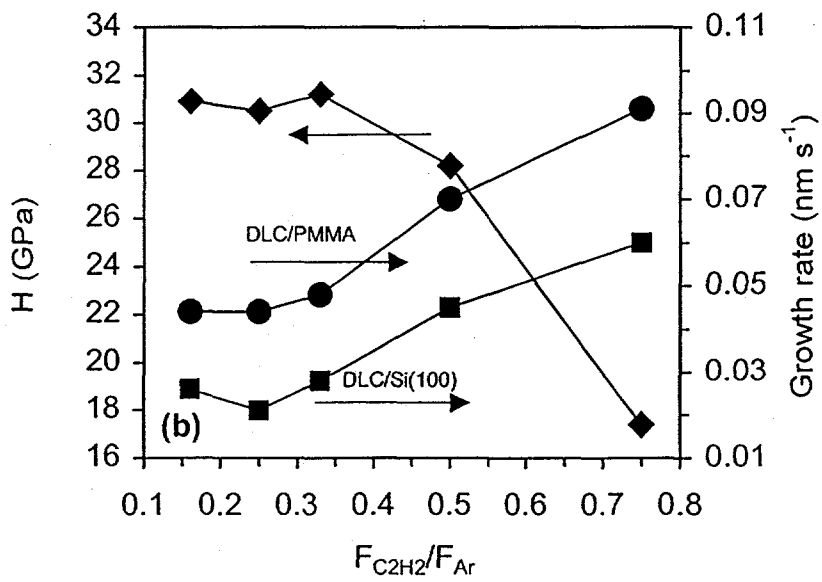
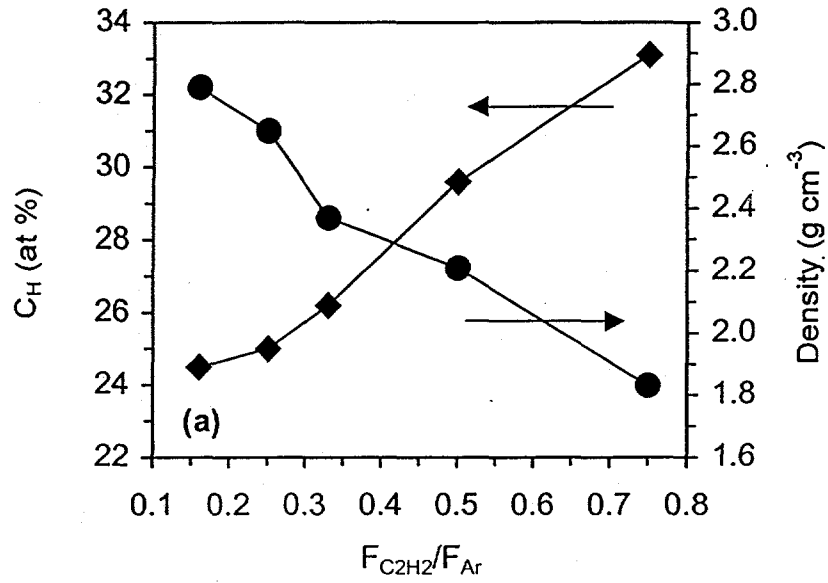


Figure 2

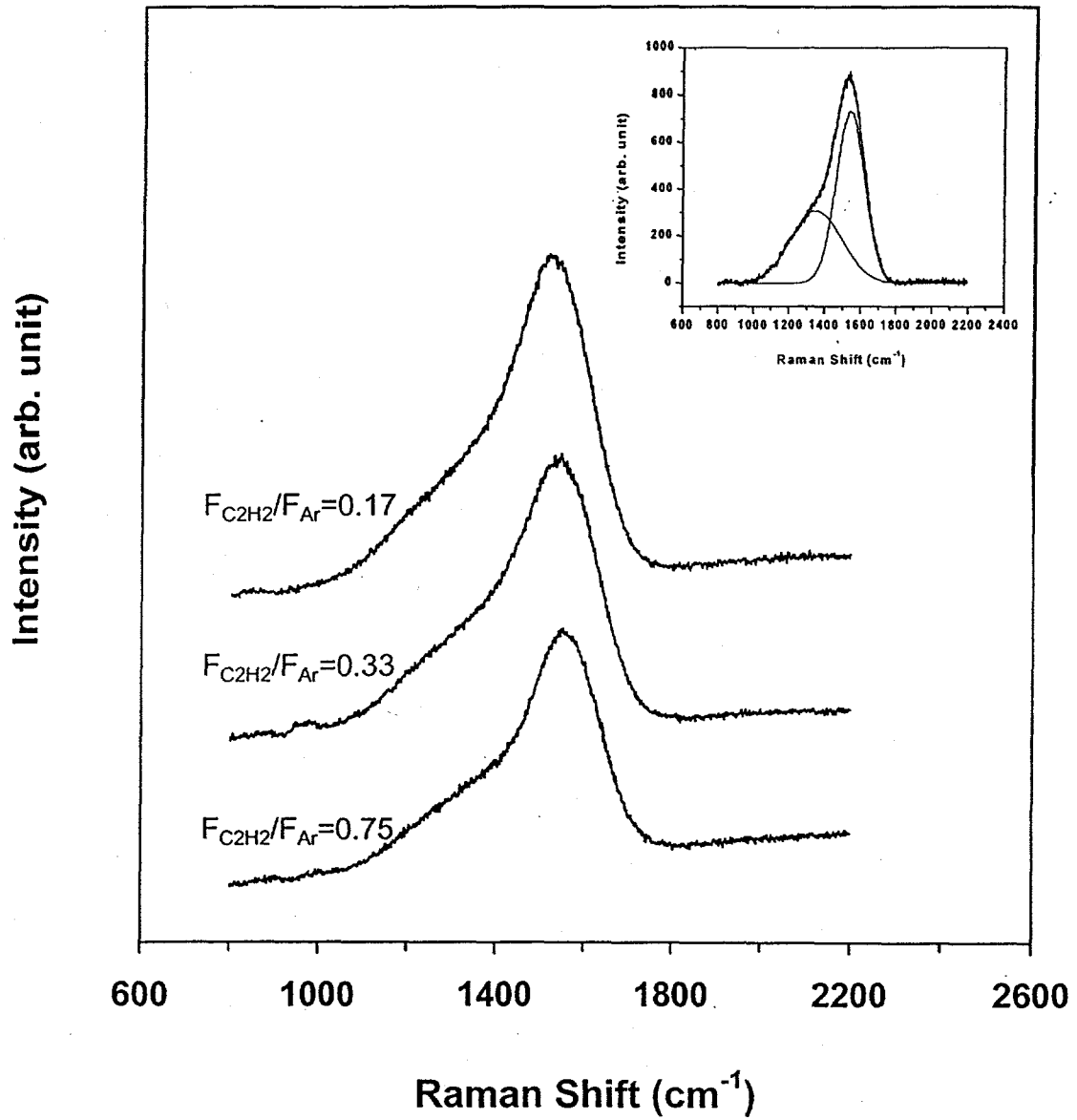


Figure 3

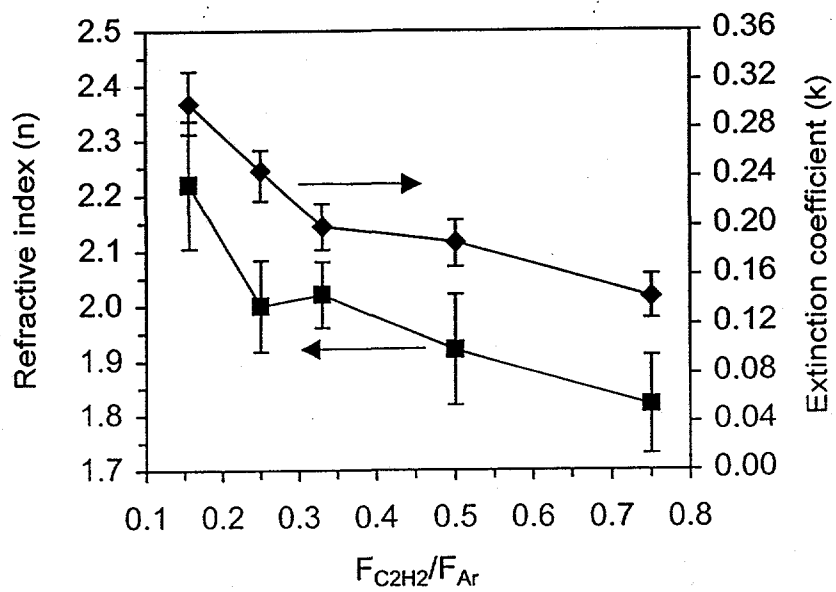


Figure 4

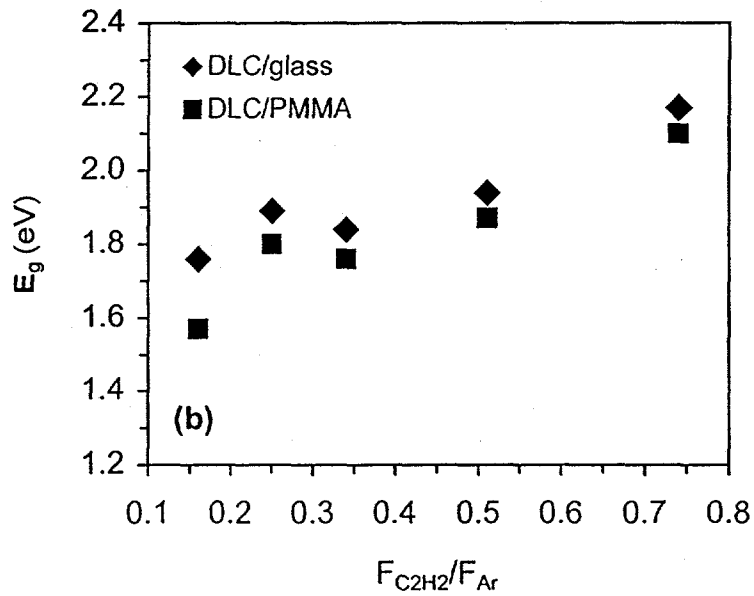
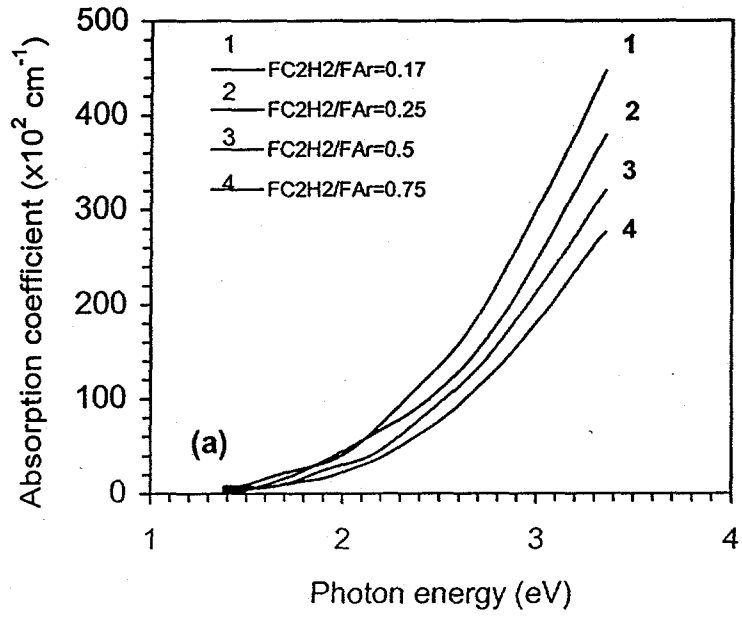


Figure 5

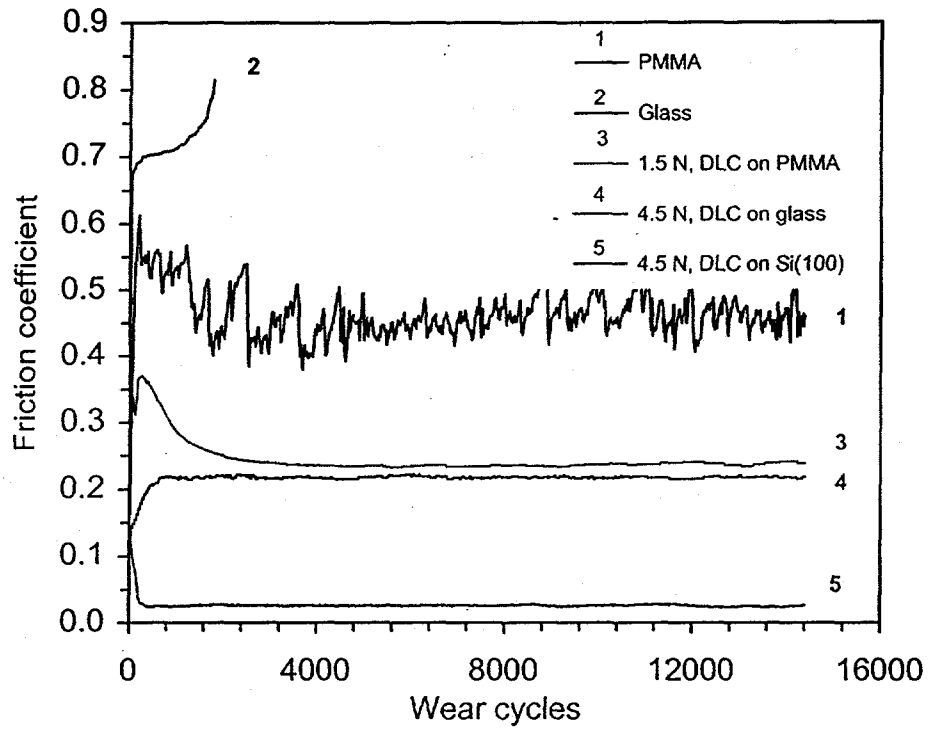


Fig. 6

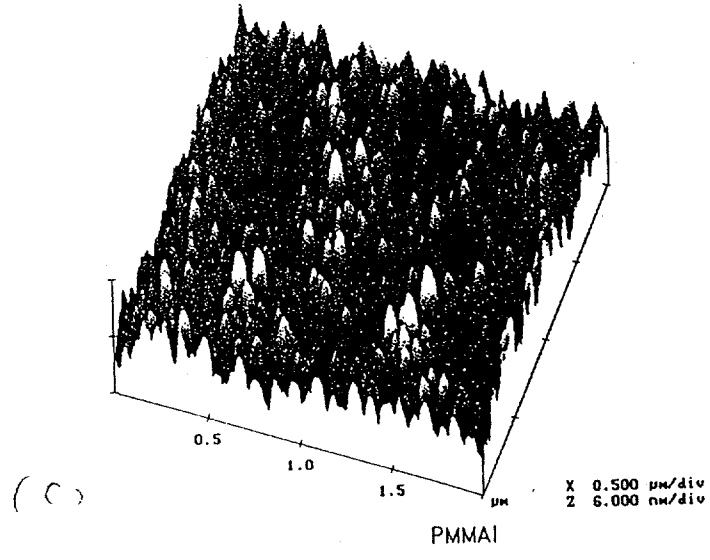
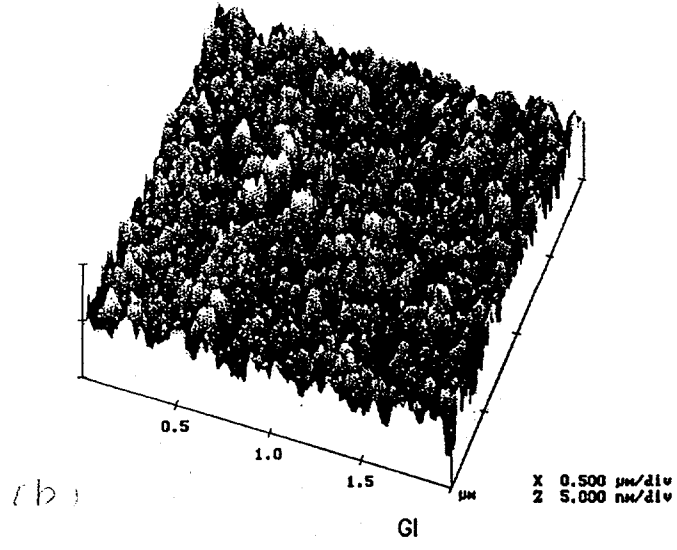
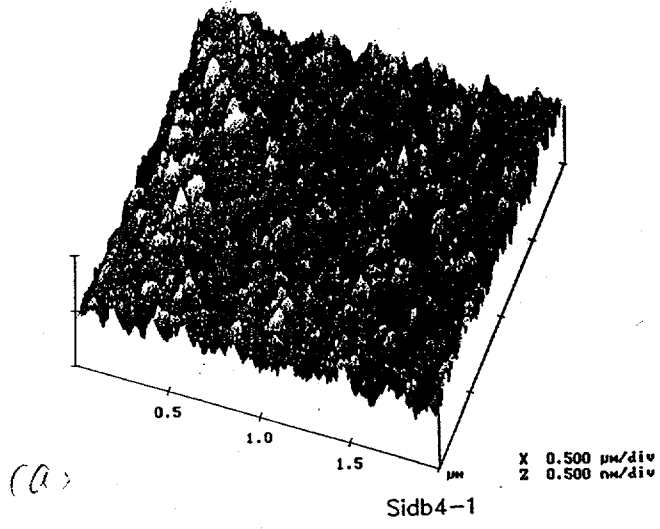


Fig. 6