Synthesis of Ag-doped hydrogenated carbon thin films by a hybrid PVD–PECVD deposition process

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Abstract. Silver-doped hydrogenated amorphous carbon (Ag-DLC) films were deposited on Si substrates using a hybrid plasma vapour deposition–plasma enhanced chemical vapour deposition (PVD–PECVD) process combining Ag target magnetron sputtering and PECVD in an Ar–CH₄ plasma. Processing parameters (working pressure, CH₄/Ar ratio and magnetron current) were varied to obtain good deposition rate and a wide variety of Ag films. Structure and bonding environment of the films were obtained from transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS) and Fourier transform infrared (FTIR) spectroscopy studies. Variation of processing parameters was found to produce Ag-doped amorphous carbon or diamond-like carbon (DLC) films with a range of characteristics with CH₄/Ar ratio exercising a dominant effect. It was pointed out that Ag concentration and deposition rate of the film increased with the increase in d.c. magnetron current. At higher Ar concentration in plasma, Ag content increased whereas deposition rate of the film decreased. FTIR study showed that the films contained a significant amount of hydrogen and, as a result of an increase in the Ag content in the hydrogenated DLC film, sp^2 bond content also increased. The TEM cross sectional studies revealed that crystalline Ag particles were formed with a size in the range of 2–4 nm throughout an amorphous DLC matrix.

Keywords. Amorphous carbon; FTIR analysis; silver; hybrid deposition; hydrogenated DLC.

1. Introduction

Nanostructured materials can exhibit unique mechanical, chemical and electronic properties and thus possess a great potential for applications in tribology, electronics, biotechnology and information technology (Wang *et al* 1998). The tribological behaviour is mainly dependent on surface characteristics of the materials. Nanocomposite films have a tremendous potential to modify and tailor the surface state, and optimize wear and frictional behaviour of the materials while maintaining desirable bulk material properties such as strength and toughness (Bull 1995).

During the past two decades, amorphous carbon or diamond-like carbon (DLC) films have attracted an overwhelming interest from both industry and the research community. DLC films offer a wide range of exceptional physical, mechanical, biomedical and tribological properties. Hydrogenated DLC films are characterized as amorphous and consist of a mixture of diamond-like and graphite-like domains. They are usually referred to as a-C:H films. The diamond-like character that gives high hardness to DLC films comes from the sp^3 bonding of C atoms, whereas the sp^2 bonding gives the graphite-like behaviour

such as low coefficient of friction (Robertson 1992). Tribological behaviour of DLC films strongly depends on both the nature of the coating and test conditions including applied load, speed, environment and contact temperature and interfacial chemistry parameters (Erdemir and Donnet 2006).

The addition of metal atoms in the carbon matrix causes great changes in chemical and tribological properties of DLC coatings and such metal alloyed coatings are often denoted as Me-DLCs. In Me-DLC composite films containing metal such as Cu or Ag inert with respect to carbon, interatomic forces in crystallite–carbon interfaces can be weak. These films may have low internal stress, high ductility and toughness. In other words, the tribological behaviour and friction properties of these Me-DLC composite films such as Cu–C or Ag–C films can be very distinct from those of carbide forming Me-DLC composite films (Pauleau and Thierry 2004; Hussain and Pal 2006). Recently, DLC-metal composite coatings, especially Ag-DLC, have gained increasing attention as a new area of DLC research. Ag-DLC composite films can be used in optical device applications (Ahmed *et al* 2009), cardiovascular devices or implants due to the surface anti-bacterial properties (Kwok *et al* 2007; Baba *et al* 2013; Lan *et al* 2013), of space devices due to wear resistance against oxygen plasma etching (Marciano *et al* 2008), for low-threshold field emitter application (Ahmed *et al* 2008),

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electrodes in electrochemical analysis in microelectromechanical systems (Lee and Ting 2003) and tribological applications (Liu and Meletis 1997; Manninen *et al* 2013; Takeno *et al* 2013).

Me-DLC films have been synthesized by different deposition methods, thereby providing a wide range of different film properties. Yu *et al* (2009) studied non-hydrogenated Ag-DLC films prepared by a mid-frequency dual-magnetron system and they investigated influence of Ag content and grain size on microstructure. They found that the Ag nanocrystallites were dispersed in the DLC matrix, and increasing Ag content resulted in an increase of the Ag nano size. Paul *et al* (2009) synthesized silver/DLC composite films by capacitively coupled plasma (CCP) chemical vapour deposition (CVD) technique. They reported that silver nanocrystallite size increased from 10 nm to 36 nm for films deposited with increasing amount of silver in the DLC matrix. Choi *et al* (2007) reported that Ag atoms into DLC films, deposited by hybrid ion beam deposition system (an end-Hall-type ion gun and d.c. magnetron sputtering), played a critical role due to the weak bond between C and Ag atoms resulting in the reduction of the residual stress.

During the synthesis of metal-doped hydrogenated DLC, working pressure, magnetron current and concentration of gaseous species in the plasma play a critical role because they all affect the properties and thickness of the film. In the present investigation, the effect of three major PVD/PECVD processing parameters was studied. Ag-DLC films synthesized by combining d.c. plasma enhanced chemical vapour deposition (PECVD) and d.c. magnetron sputtering of Ag target at different gas mixtures, magnetron current and working pressures. The films, which had a regular increment of Ag content and good deposition rate, were characterized using Fourier transform infrared (FTIR) spectroscopy and high-resolution transmission electron microscopy.

2. Experimental

All thin films were deposited on Si (100) wafers with an area of 2 cm². Prior to deposition, the wafers were cleaned in acetone and then cleaned with compressed air. The hybrid PVD/PECVD deposition system, which has been used for the synthesis of Ag-DLC, is seen in figure 1. The chamber was roughed down to 20 mTorr using the mechanical pump, and then the chamber was exposed to the cryopump by opening the gate valve. The base pressure of the chamber was around 2.5×10^{-6} Torr. The chamber was purged two times with Argon prior to any processing. The substrate was sputtered cleaned with Argon at a bias of -1000 V for 10 min at a pressure of 30 mTorr. Sputter cleaning removes any surface contamination and contributes to good adhesion of the films.

Hydrogenated Ag-DLC films were deposited by d.c. sputtering of Ag with a CH₄/Ar mixture for a d.c. negative bias (-1000 V) during 30 and 60 min. Ag was initially pre-sputtered on the shutter plate to establish a good plasma, and the shutter was opened when a proper plasma was

Figure 1. Hybrid PVD/PECVD deposition system.

established. The working pressure (20, 30 and 40 mTorr), CH₄/Ar ratio (10/90–30/70) and magnetron current (30–100 mA) were varied to obtain good deposition rate and a wide variety of Ag films. Pure DLC films were also prepared using the hydrocarbon ion beam without operating the sputter gun. Two small areas on the opposite sides of each sample were masked both to prevent deposition and to allow film thickness measurement. The surface optical profilometer (WYKO NT 1000), which functions on the principle of light interference at a magnification of 10X, traverses on the masked area and measures the height difference. At least twelve measurements were performed for each masked area, and the average values of the film thickness were reported. The deposition rate of Ag-DLC films was estimated by the ratio of the observed thickness to the total deposition time. Scanning Electron Microscopy (JEOL JSM 6100) in conjunction with Energy Dispersive Spectroscopy (Oxford, model 7582) system was used to obtain the composition of the Ag-DLC films (Ag, C). The samples were analyzed at an acceleration voltage of 20 keV. EDS analysis values signified the average of at least twelve measurements.

The investigation of C–H bonding in the DLC and Ag-DLC films (Ag-DLC30-3, Ag-DLC30-7, Ag-DLC30-9, Ag-DLC30-10 and Ag-DLC30-11) was performed by FTIR spectroscopy (Nicolet) in the range 2800–3150 cm⁻¹ which reveals a high H content in the films. The spectrum of the films was subtracted from the Si wafer background to obtain the absorbance response of the films. The resultant curve was smoothed twice using a 25 point smoothing in the OMNIC software. The region for C–H stretching was selected and subjected to a base line correction. The corrected curve was subjected to Fourier self deconvolution to get the component peaks. Deconvolution of the spectra was performed using Gaussian distribution to calculate the C–H $sp^3/(sp^2 + sp^3)$ ratio. HRTEM of Ag-DLC samples was performed on Hitachi H-9500 HRTEM operated at 300 keV with a point-to-point resolution of 0.18 nm.

3. Results and discussion

3.1 Synthesis of Ag-doped hydrogenated DLC thin films

A series of experiments were performed to obtain a varying Ag content in DLC. The deposition parameters, obtained film thickness, deposition rate and Ag content of Ag-DLC thin films for -1000 V bias voltages are given in table 1. It was observed that the working pressure and CH_4/Ar ratio played a very important role for controlling the Ag content in DLC. As seen in table 1, silver was obtained in the DLC up to 8.6 at% at 100 mA target current and 10/90 of CH_4/Ar ratio for 30 mTorr working pressure. The films obtained in this working pressure had a uniform colour and appearance. In the case of 20 mTorr working pressure, the film quality was not good (not uniform colour) and adherent to substrate although higher Ag content was found 10.28 and 13.7 at% for CH_4/Ar ratios of 15/85 and 10/90, respectively. The deposition rate changed with the percentage of CH_4 , and the highest deposition rate value was found for 30% CH_4 .

Figure 2 shows the effect of methane flow in the gas mixture composed of Ar and methane on deposition rate and Ag content in the DLC for 40, 30 and 20 mTorr working pressures and -1000 V bias voltages. Although Ar gas ratio was increased to 90%, very little silver could be found in the DLC because of a significant poisoning of the target for the samples carried out at 40 mTorr. As seen in figure 2(b), the Ag content in the hydrogenated DLC decreased with an increase in methane flow in the gas mixture. However, the deposition rate of nanocomposite DLC increased with an increase in methane flow percentage. When the Ar concentration was less than 90%, the incorporation of Ag in the DLC film was

detected slightly. This was probably due to a significant poisoning of the target caused by the high flux of carbon ions that decreases the sputtering yield of the silver target. Similar target poisoning observations were reported for metal incorporated DLC films synthesized in various deposition systems (Bewilogua *et al* 2000; Choi *et al* 2008a, b; Wang *et al* 2011).

As the CH_4 flow decreased, the silver content in the DLC increased and the deposition rate decreased at 20 mTorr (figure 2c). As seen in figure 2(c), the deposition rates of the films coated with 10% and 15% methane flows were higher than that of films coated with 20% methane flow. This can be attributed to a higher silver content of the samples treated in 10% and 15% methane flow ratios. It was reported that a higher Ar/ CH_4 ratio leads to a higher metallic composition or a lower carbon composition (Wu and Ting 2002; Nakao 2009). Therefore, it is believed that a sufficient amount of metallic constituents is required to allow the metal to form crystalline structure. The Ar/ CH_4 ratio also affects the surface morphology of the Me-DLC thin films.

The optimum pressure and Ar/ CH_4 ratio for the synthesis of Ag-DLC were selected as 30 mTorr and 10/90 ratio, respectively for obtaining a uniform colour of the film and avoiding of target poisoning. Variation in Ag content was achieved by varying the magnetron current (table 1). Figure 3 indicates the effect of d.c. current on deposition rate and Ag content in the DLC for 30 mTorr working pressure and 10% methane flow. As seen in figure 3, the deposition rate increased abruptly up to 80 mA. After this current, the deposition rate was almost stable then it increased slowly with an increase in d.c. current. However, Ag content in the DLC increased almost linearly with an increase in d.c. current.

Table 1. Variation of film thickness, deposition rate and Ag content of Ag-DLC thin films with gas ratio and d.c. power and working pressure for -1000 V bias voltage.

Samples	Working pressure (mTorr)	CH_4/Ar ratio	d.c. Power to target (mA)	Film thickness (nm)	Deposition rate (nm/min)	Ag content (at%)
DLC	40	83/17	–	7806	6.5	–
Ag-DLC40-1	40	30/70	100	490 ± 4.2	16.33	0.5 ± 0.05
Ag-DLC40-2	40	20/80	100	435 ± 5.3	14.5	0.5 ± 0.04
Ag-DLC40-3	40	10/90	100	60 ± 2.2	2	0.5 ± 0.06
Ag-DLC30-1	30	30/70	100	340 ± 6.3	11.33	1 ± 0.19
Ag-DLC30-2	30	20/80	100	305 ± 3.6	10.17	0.8 ± 0.25
Ag-DLC30-3	30	15/85	100	180 ± 4.3	6	2 ± 0.31
Ag-DLC30-4	30	10/90	100	160 ± 2.3	5.33	8.6 ± 0.37
Ag-DLC30-5	30	10/90	50	100 ± 3.4	1.66	2.4 ± 0.45
Ag-DLC30-6	30	10/90	70	105 ± 4.6	1.75	2.16 ± 0.48
Ag-DLC30-7	30	10/90	80	215 ± 6.7	3.58	3 ± 0.42
Ag-DLC30-8	30	10/90	85	223 ± 5.3	3.72	3.23 ± 0.47
Ag-DLC30-9	30	10/90	90	214 ± 4.6	3.56	3.7 ± 0.45
Ag-DLC30-10	30	10/90	95	226 ± 6.7	3.76	5.7 ± 0.29
Ag-DLC30-11	30	10/90	100	253 ± 5.5	4.22	8.3 ± 0.46
Ag-DLC20-1	20	30/70	100	275 ± 7.8	9.16	0.8 ± 0.1
Ag-DLC20-2	20	20/80	100	104 ± 6.8	3.47	1.5 ± 0.15
Ag-DLC20-3	20	15/85	100	220 ± 6.3	3.66	10.28 ± 0.55
Ag-DLC20-4	20	10/90	100	124 ± 5.8	4.13	13.7 ± 0.58

Figure 2. Effect of methane flow in the gas mixture composed of Ar and methane on deposition rate and Ag content of Ag-DLC films at bias voltage of -1000 V for (a) 40 mTorr, (b) 30 mTorr and (c) 20 mTorr working pressure.

Figure 4(a) and (b) indicates the deposition rate and Ag content changes in the DLC films as a function of working pressure for 10%, 20% and 30% methane flows. As the working pressure increased, the deposition rate of Ag-DLC also increased. However, as seen in figure 4(a), the deposition rate did not have the same trend for 10% methane flow. This can be attributed to a problem for 40 mTorr. Choi *et al* (2008a, b) found that the deposition rate of DLC coatings linearly increased with increasing deposition pressure. Additionally, it is reported that radical species are increased with increasing pressure. This suggests that the plasma density is increased so that the ion flux is also increased in amount at

Figure 3. Effect of d.c. current on the deposition rate and Ag content in the Ag-DLC films for 30 mTorr working pressure and 10% methane flow.

Figure 4. (a) Deposition rate and (b) Ag content variation in the a-C:H films as a function of working pressure for 10%, 20% and 30% methane flows.

the same time. Therefore, it is reasonable that the deposition rate is increased according to the increase of pressure. Furthermore, Gupta *et al* (2007) reported that the deposition rate increased at higher pressures due to larger hydrocarbon flux reaching the substrate surface. Ag content of Ag-DLC films decreased with an increase in methane flow as shown

Figure 5. Comparison of FTIR spectra obtained from the DLC and Ag-DLC thin films.

in figure 4(b). The highest Ag content was observed for 10% methane flow due to the fact that a significant poisoning of the target was caused by the high flux of carbon ions for 20% and 30% methane flow.

3.2 FTIR and HRTEM analysis of Ag-DLC thin films

FTIR is a widely used technique to characterize C–H bonding in hydrogenated DLC (a-C:H) films. Bonding properties of hydrogenated DLC films can be changed by introducing the second phase such as Ag. FTIR spectrum analysis was used to investigate the change and the presence of sp^2 and sp^3 hybridization in the atomic bonding structure between carbon and hydrogen in the hydrogenated DLC film. The samples, DLC, Ag-DLC30-3 (2 at% Ag), Ag-DLC30-7 (3 at% Ag), Ag-DLC30-9 (3.7 at% Ag), Ag-DLC30-10 (5.7 at% Ag), Ag-DLC30-11 (8.3 at% Ag) were selected for FTIR analysis. Figure 5 shows FTIR spectra obtained from the DLC and Ag-DLC thin films. Generally, the vibration mode of the FTIR spectrum obtained from DLC films is classified into stretching-type and deformation-type bands. The vibration mode of the stretching-type carbon–hydrogen bond is observed in the range of 2850–3300 cm^{-1} and that of the deformation-type is observed in the range of 1300–1700 cm^{-1} . In our investigation, only the stretching-type vibration mode was observed for the DLC films. The C–H stretching mode is revealed in the range of 2850–3300 cm^{-1} of the FTIR absorption spectrum. The CH_3 asymmetric stretching vibration occurs at around 2975–2950 cm^{-1} , whereas the absorption forms at about 2930 cm^{-1} for CH_2 . The CH_3 symmetric vibration and the CH_2 absorption occur

in the ranges of 2865–2885 and 2870–2840 cm^{-1} , respectively (Heitz *et al* 1998). As seen in figure 5, after wavenumber of 2950 cm^{-1} , absorption peak positions shifted to lower wavenumbers with an increase in Ag content in the DLC. Paul *et al* (2009) reported that by introducing Ag in DLC, the peak related to Ag–H bonds became broad and shifted to a lower wavenumber. Furthermore, Hussain and Pal (2007) also indicated that the FTIR spectrum for DLC got significantly modulated by the incorporation of nanocrystalline Ag–Cu.

Deconvoluted FTIR spectrums of C–H stretch region and the various bonding states of the C–H stretching (as a table) in the DLC films are shown in figure 6(a–c). The relative amount of each bonding type was obtained by estimating the area under each peak. As seen in figure 6(a–c), by introducing Ag in the DLC, the shoulders at ~ 2800 –2850 cm^{-1} and ~ 2970 –3150 cm^{-1} , which were arisen due to sp^3 -CH symmetric and sp^2 -CH olefinic stretching vibrations, which were arisen, became prominent, respectively. The obvious absorption peaks at 2830–2920 and 2960–2980 cm^{-1} are due to the hydrogen atom in the CH_2 (sym-asym)/ CH_3 (sym) stretching bands, all bonded to sp^3 -type carbon. The peaks located at 2950 cm^{-1} and in the range of 3000–3110 cm^{-1} may be assigned to sp^2 - CH_2 (sym) and sp^2 -CH (olef./arom.) bonding stretching modes (Mutsukura *et al* 1992; Heitz *et al* 1998; Son *et al* 2001). Teii (1998) observed relative increases in the intensity of the sp^2 bond at 3000–3150 cm^{-1} . From these results, it can be said that the bonding structures between carbon and hydrogen in the Ag-DLC films consist of sp^3 and sp^2 bonds. Figure 7 shows variation of $sp^3/(sp^2 + sp^3)$ ratio based on C–H bonds with

Figure 6. Deconvolution of the C–H stretch and FTIR vibrational mode assignment in the C–H stretch region (as a table) for (a) DLC, (b) Ag-DLC30-7 containing 3 at% Ag and (c) Ag-DLC30-11 containing 8.3 at% Ag thin films.

Ag content in the DLC films. It can be seen in figure 7 that, as the Ag content in the DLC increased, the sp^3 bond decreased. The ratios of $sp^3/(sp^2 + sp^3)$ were obtained from

the deconvoluted spectra based on the areas under the sp^2 and sp^3 peaks. The ratio of undoped DLC film was 0.916, whereas $sp^3/(sp^2 + sp^3)$ ratios of Ag-DLC30-7 (3 at%)

Figure 7. Variation of $sp^3/(sp^2+sp^3)$ ratio based on C–H bonds with Ag content in the DLC films.

Figure 8. HRTEM images of (a) cross-sectioned Ag-DLC30-7 film containing 3 at% Ag (b) nanocrystalline Ag grains under higher magnification of in (a). The arrows show crystalline Ag particles.

and Ag-DLC30-11 (8.3 at%) films were 0.802 and 0.422, respectively. Paul *et al* (2009) observed that sp^2/sp^3 ratio derived from deconvoluted FTIR spectra increased from 0.03 for the film lesser Ag content to 0.21 for films with higher Ag content in the DLC matrix. It may thus be inferred that the Ag-DLC films became sp^2 rich with increasing incorporation of silver nano-crystallites. Similar trends have been observed for other metal-doped DLC films (Klages and Memming 1989; Corbella *et al* 2004; Pandey and Hussain 2011).

Figure 8(a and b) shows HRTEM cross-section microstructure of DLC film containing 3 at% Ag. HRTEM analysis in figure 8a has revealed that silver nanoparticles were distributed uniformly throughout the film. The darker spots (indicated by arrows in figure 8b) correspond to the metallic Ag region, while the brighter region corresponds to the amorphous carbon matrix. As seen in figure 8(b), Ag-incorporated DLC film showed amorphous carbon structure and also partially segregated and formed crystalline Ag particles with a size in the range of 2–4 nm. It can be clearly seen that there are ordered areas with different orientation of crystalline planes within the dark spots with nearly spherical shape.

4. Conclusions

A hybrid deposition system composed of d.c. plasma enhanced chemical vapour deposition (PECVD) and d.c. magnetron sputtering was successfully employed to deposit Ag-incorporated hydrogenated-DLC films. It was observed that the working pressure and CH_4/Ar ratio played a very important role in controlling the Ag content in DLC. When the Ar concentration was less than 90%, the incorporation of Ag in the film was detected a little probably due to a significant poisoning of the target. For this study, the optimum pressure and Ar/CH_4 ratio for the synthesis of Ag-DLC were 30 mTorr and 10/90, respectively, because of for obtaining a uniform film colour and avoiding of target poisoning.

FTIR spectra indicated that The C–H stretching mode was revealed in the range of $2850\text{--}3100\text{ cm}^{-1}$. FTIR results showed that the bonding structure between carbon and hydrogen in the DLC film consisted mostly of sp^3 and a few sp^2 bonds. As the Ag content in the DLC increased, the sp^3 bond decreased. HRTEM observation showed that the films contained uniformly distributed Ag nanoparticles in the range of 2–4 nm size in an amorphous DLC matrix.

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