CHAPTER 5
CONCLUSIONS

5.1 Introduction

In this project, the electrical characteristics of the planar coil radio frequency inductively coupled plasma (PC-RFICP) system constructed are determined. The PC-RFICP system is then used for plasma material processing. The applications of the PC-RFICP for plasma material processing that have been tested include nitriding of stainless steel and titanium surfaces, PECVD of titanium nitride film, diamond-like carbon film and hydrogenated amorphous carbon film. The following sections are conclusions obtained from the characterization of the PC-RFICP system, as well as studies conducted on the applications of using the PC-RFICP system for plasma material processing mentioned above. Suggestions to upgrade the current PC-RFICP system to improve the efficiency of the nitriding process and to obtain film with better and reproducible quality are also presented.

5.2 Electrical characteristics of the Planar Coil Radio Frequency Inductively Coupled Plasma (PC-RFICP)

The planar coil PC-RFICP system is powered by a 550W, 13.56 MHz RF generator. The RF generator is coupled to the load via a matching network consisting of a step down transformer and variable vacuum capacitor. The variable vacuum capacitor is used to tune the circuit in order to transfer maximum power to the plasma. The efficiency in power transferred (as indicated by the quantity of coil current) of the “home built” matching network (LC resonant circuit) is comparable to the automatic impedance matching network unit (ENI model MW-5D).

E to H mode transition in argon plasma, argon/nitrogen, hydrogen plasma, and methane/hydrogen admixture plasma has been studied at different pressure and different power for the planar coil RFICP system. The pure argon RF plasma is able to transit from the E to H mode plasma at lower RF power compare to argon-nitrogen, hydrogen and methane-hydrogen admixture plasma. The sudden changes in the emission intensity of plasma and coil current have been observed during the E to H mode transition. The hysteresis behavior typically observed in an inductively coupled plasma mode transition is also observed. The coil current for the H-mode plasma is lower than the E-mode plasma due to an increase in the plasma current indicating that the H-mode discharge has higher power coupling efficiency. In the H-mode discharge, there is almost no increase in the coil current as the RF power is increased, indicating that the extra energy is absorbed by the plasma for plasma expansion (as observed visually).

The effect of confining the RF plasma into a small volume using a glass funnel on the E to H mode transition, and the induction heating effect by the eddy current induced on the substrate in the PC-RFICP system are two phenomenal effects found in this system. The confinement of the RF plasma into a small volume within a glass funnel is able to reduce the RF power required for E to H mode discharge transition. The induction heating effect of silicon substrate by the eddy current that is developed on the substrate is able to heat up the substrate to 800 to 900^{o}C. However, it is also demonstrated that the RF power available in the present system is not sufficient to transit into a H mode discharge when induction heating of the silicon substrate occurs since a portion of the RF power is used to induce the eddy current on the silicon substrate.

Langmuir probe measurement is conducted on the plasma generated by the PC-RFICP system for the pure argon and argon/acetylene admixture discharges to obtain the ion density (n_i) and electron temperature (T_e). The values of T_e obtained for the pure argon discharge under all conditions are in the range of 0.25 to 8.5 eV, while the values of n_i measured is in the order of 10^{17} m^{-3}. On the other hand, the values of T_e obtained from the argon/acetylene admixture discharge under all conditions are in the range of 3 to 5 eV, while n_i measured is in the order of 10^{16} m^{-3}. For the pure argon discharge, the values of n_i are observed to have an obvious increase with applied RF power after the discharge has transit to the H mode of operation. For the argon/acetylene admixture discharge, the values of n_i are observed to increase with applied RF power. Both the pure argon and argon/acetylene admixture discharges show a decrease in n_i with the increase in discharge pressure. The values of T_e for the pure argon discharge show a decrease with the increase in RF power (due to the increase in n_i). However, the values of T_e for the argon/acetylene admixture discharge appear to increase with the increase in RF power. The applied RF powers used to sustain the argon/acetylene admixture discharge facilitate the ionization of electrically neutral species (argon atoms and acetylene molecules) by colliding with energetic electrons (heated by the RF power). The increasing RF powers supplied also appear to provide “extra” energy to the electrons to “overcome” the increasing electron-ion collisions frequency, due to increasing n_i, when RF power is increased, which may lower the electron temperature. The lower ion density in the “environment” of the argon/acetylene admixture discharge compared to the pure argon discharge also appear to assist the more effective heating of the electrons by the RF power. Hence, an increase in n_i,and T_e when the applied RF power is increased. The stability and reproducibility of plasma generated by the PC-RFICP system that is developed in this work allows it to have the versatility for plasma material processing.

5.3 Nitriding of metal surfaces using the Planar Coil Radio Frequency Inductively Coupled Plasma System

The planar coil PC-RFICP system using the automatic Π matching network is used to perform nitriding on AISI 316 stainless steel substrate, in nitrogen/hydrogen admixture plasma with gas ratio of 9:1. The thickness of the nitrided layer measured from cross-sectional view of the stainless steel substrate is 10 ± 2 μm after the nitriding time of 2 hours. This measurement was confirmed using EDAX analysis scanned across the transverse section of the substrate cross section. GAXRD scan of the nitriding layer show a dominant ε-$(Fe,Cr)_3N$ phase present in the nitrided layer. Vickers microhardness test on the nitriding layer give a surface hardness of 1365±300 Hv as compared to unnitrided surface which has a hardness of 186±8 Hv. The nitrided AISI 316 stainless steel substrate showed an increase in hardness of about 7 times.

Nitriding of titanium substrate using argon/nitrogen admixture using the PC-RFICP system with the LC resonant circuit as the matching network is demonstrated. As in the case of nitriding of stainless steel, the application of substrate bias is proven beneficial in aiding the nitriding process. At lower bias voltage (0 to –500 V), the efficiency of nitriding is generally lower. The desired δ-TiN phase is formed when nitriding is done at higher bias voltage like -1000 V and –1500 V. However, nitriding at –1500 V is accompanied by an undesirable microstructural change of the titanium.

Nitriding of titanium by RF plasma has also improved the mechanical and tribological properties of titanium. The nitrided titanium at bias voltages of -1000 V and –1500 shows more than 4 times improvement in surface hardness compared to the untreated titanium and the wear resistance properties of titanium has also been improved. The surface roughness of nitrided titanium has been demonstrated to be a function of the bias voltage or ion bombardment intensity.

5.4 Plasma enhanced physical vapor deposition (PEPVD) of titanium nitride film on stainless steel substrate

The planar coil radio frequency inductively coupled plasma system with LC resonant circuit as the matching network has also been used for plasma enhanced physical vapor deposition of titanium nitride on stainless steel samples. The plasma enhanced physical vapor deposition technique involves the sputtering of the pure titanium target immersed in the H-mode plasma. The intention of immersing the titanium target in the H-mode plasma is to achieve higher sputtering rate and hence faster deposition rate. Titanium nitride thin films, which appear as golden yellow films are successfully deposited on stainless steel plates.

The titanium nitride thin film deposited onto stainless steel substrates through the PEPVD technique is dependent on deposition process parameters, especially substrate bias voltage, which may influence TiN film composition, surface morphology, and structure.

The color of titanium nitride obtained from the experiments, which determine the “purity” of the TiN film is dependent on the substrate bias voltage that is applied during the deposition process. The change in the color of the coated TiN films may be due to the change in the composition of the TiN film, especially that of oxygen. The color of the TiN changes from golden yellow to dark brown when the percentage of oxygen in the film becomes higher. At lower substrate bias voltage (0 to –40 V), the percentage of oxygen is found to be higher and this causes the color of the TiN film to become dark brown.

Sufficiently high substrate bias voltage (-80 V to -160 V) is able to lower the oxygen content in the film. The atomic percentage of oxygen drops from 30.72 % at 0 V to 2.13 % at -80 V.

The atomic percentage of nitrogen is always higher than titanium in the range of substrate bias voltage used in this study. The ratio of titanium to nitrogen is found to be around 0.6 for the operated bias voltage in the range of -60V to -160V. However, this ratio is not a true reflection of the TiN film composition since a certain amount of the measured nitrogen may come from the nitrided layer of the stainless steel substrate, which may have been formed during the deposition process.

XRD analysis shows that the TiN films deposited with the substrate bias voltage from –40 V to –160 V contain crystalline titanium nitride. However, at substrate bias voltage from 0 V and –20 V, the peaks observed are determined to be zeta titanium nitride (Ti_4N_{3-x}), which appears as black color compound on the substrate.

The film prepared at negative substrate bias voltage higher than -60 V appears to have a denser and smooth surface due to enhancement in the surface mobility of deposited atoms. The roughness of the film surface is kept below 10 nm for the bias voltage higher than -60 V. Below -60 V surface roughness increases to 49.6 nm at -40 V, 91.5 nm at -20 V and 82.2 nm at 0 V.

There is no change in the film appearance within the range of the operating pressure of 0.5 to 3.5 mbar. All the films deposited within the stated pressure range demonstrated the typical golden yellow color characteristic of TiN film.

The change in pressure within the range gives no effect to the atomic percentages of nitrogen and oxygen. Nevertheless, the atomic percentage of titanium drops gradually from 38.19 % at 0.50 mbar to 12.52 % at 3.50 mbar. This gradual drop is mainly caused by lower deposition rate at higher pressure.

For the operating pressure between 0.50 to 3.50 mbar, XRD analysis shows that all the coatings obtained have similar peaks of titanium nitride and stainless steel. Titanium nitride peak with lattice plane of (200) is the highest in intensity indicating that this is the preferred plane for most of the crystals formed. There is a change of orientation plane from (222) to (111) and (311) as the pressure is increased.

When no substrate bias voltage is applied, two peaks due to titanium oxy nitride ($TiO_{0.34}N_{0.74}$) are detected for the sample deposited without hydrogen. However, titanium oxy nitride peak is not seen in TiN films deposited in the presence of hydrogen gas. The TiN peaks detected for conditions with, and without hydrogen gas are due to zeta-titanium nitride (Ti_4N_{3-x}) and titanium nitride (Ti_2N).

When the substrate is biased at -100 V, no $TiO_{0.34}N_{0.74}$, Ti_4N_{3-x} or Ti_2N peaks are observed for both conditions with and without hydrogen gas indicating that the effect of the substrate bias voltage is more dominant than the effect due to hydrogen gas.

5.5 Plasma enhanced chemical vapor deposition of (PECVD) of diamond-like carbon films on silicon substrates

PECVD of diamond-like carbon films on silicon substrates is conducted in a plasma of methane and hydrogen admixture gas using the planar coil radio frequency inductively coupled plasma system with LC resonant circuit as the matching network. The different percentages of methane mixed with hydrogen have significant effect on the texture of the surface morphology of the diamond-like carbon thin film. Besides, the pretreatment of the diamond paste on the silicon substrate seems to improve the nucleation of the diamond-like carbon film.

The silicon substrate that is placed away from the planar coil so as to avoid the induction heating effect does not show any nucleation, even though it has been grazed with diamond paste. The observation shows that induction heating which is able to heat up the sample to temperature between 800-900^{o}C is vital for the nucleation and growth of diamond-like carbon film. Hence, scratching the silicon with diamond paste, and exposing the silicon to the induction heating in the plasma are crucial for nucleation, and growth of diamond-like carbon film. However, in the current PC-RFICP system, the induction heating process is not able to spread the elevated temperature uniformly, consequently the diamond-like carbon film coated on the surface is not uniform.

5.6 Plasma enhanced chemical vapor deposition (PECVD) of hydrogenated amorphous carbon (a-C:H) film

The a-C:H film deposited from admixture of acetylene/argon gas using the PC-RFICP system is polymeric and soft. From SEM micrograph analysis, the film deposited onto glass and silicon substrates appear to be homogenous. AFM imaging suggests that the type of growth of the a-C:H film during film deposition is columnar growth. The thickness of the film ranges from 0.45 μm to 1.2 μm, depending on the conditions that is used for deposition. Adhesion of the film to the substrate remains a challenging issue to be addressed as the film is observed to peel off from the substrate after prolong period of exposure to air.

The a-C:H film is found to undergo oxidation when kept under ambient condition. The oxidation of the film may be due to the presence of -C=C- group that is embedded in the microstructure of the film. The -C=C- group is reactive towards oxygen in the air forming -C=O and -OH groups as evident in the FT-IR spectrum.

The rate of the deposition of the a-C:H film is found to increase with the increase in applied RF power and discharge pressure. However, the rate of deposition decreases with increase in the flow rate of acetylene and substrate's distance from the planar coil.

The effect of the deposition parameters on optical energy gap (E_g), Urbach energy (E_u) and refractive index, n, of the a-C:H is investigated. The range of the optical energy gap that is determined for the a-C:H films deposited under different deposition parameters are between 2 to 3.6 eV. The range of the Urbach energy for the a-C:H films deposited under different deposition parameters are between 120 to 310 meV. The values of the refractive index of the a-C:H films are in the range of 1.5 to 2.0. The increase or decrease in the E_g is suggested to be related to the sp^2 fraction in the film. Eg is expected to increase when sp^2 fraction in the film decreases and vice versa. An increase in the sp^2 fraction is associated with the increase in E_u, since the latter is commonly used to measure disorder in the a-C:H film. The a-C:H film is characterized by sp^2 C clusters embedded in sp^3 C matrix. The smaller the fraction or size of the sp^2 C clusters, the more "orderly" is the a-C:H film. The increase in the n value is associated with the increase in the degree of the cross linking, density and a more compact microstructure of the film.

The microstructure of the a-C:H film are investigated using Fourier Transform-Infrared (FT-IR) Spectroscopy. The bound H content, (in at. %), and sp^3 to sp^2 ratio are determined for films deposited under different deposition parameters. The variation in the H content and sp^3 to sp^2 ratio is related to the effect of the deposition parameters on the surface processes during film deposition. The decrease in H content is related to H atom abstraction at the surface of the film during deposition. The decrease in the sp^3 to sp^2 ratio is due to the increase in sp^2 C species contributed by the -C=C- "fragments" which is in turn due to their incorporation into the matrix of the a-C:H film during deposition. As mentioned earlier, the C in the matrix of the a-C:H film is mainly of the sp^3 C species. The surface processes which contribute to the microstructure of the a-C:H film during film deposition are affected by deposition parameters such as applied RF power, discharge pressure, acetylene flow rate and the distance of the planar coil from the substrate.

Post deposition annealing of the a-C:H film in air at 250 ^{o}C is conducted. The golden yellow color of the a-C:H film is observed to turn to dark brown after annealing. The thickness of the film is also found to decrease significantly after annealing. Micro-Raman spectroscopy analysis shows the prominent appearance of the broad D and G bands which are not easily obtained by this analysis before annealing. The broad D and G bands indicate a significant increase in the graphitic nature of the film after annealing. In other words, the microstructure of the film is significantly changed. This is supported by FTIR spectrum which shows the significant increase in the sp^2 C=C vibration band at

1722 cm^{-1} and the disappearance of the C-H stretching band at ~ 3000 cm^{-1}. The annealing of the a-C:H film can reduce the magnitude of the stress in the film by removing H content in the film, and hence improving the adhesion of the film. Thus, annealing appears to be a possible method to improve the adhesion of the film, but the microstructure of the film has to be compromised.

The a-C:H film demonstrates ohmic characteristic in their electrical property. The conductivity of the film is in the range of 3.0 X 10^{-8} to 4.7 X 10^{-8} Scm^{-2}.

5.7 Suggestions for Future Work

For the purpose of plasma nitriding of metal surfaces, the currently used DC bias voltage can be replaced by high voltage pulsed DC biasing. Through biasing at high voltage, nitrogen can be implanted into the metallic substrate more easily and hence increases the efficiency of plasma nitriding.

For plasma sputter deposition process improvements can be made to the present PC- RFICP system. A temperature controllable substrate table can be designed and installed in the system to control the substrate temperature. A target holder with cooling system can be designed to prevent heating of the target by the sputtering process. High temperature produced by the sputtering process can give rise to property change to the target.

The heating of the substrate to high temperature by the induced eddy current, which is a phenomenal discovery in the PC-RFICP system, is harness for the PECVD of diamond-like carbon film. However, the heating of the substrate is not uniform resulting in the film deposited to be non-uniform. In addition to this, the magnitude of eddy current induced is dependent on the resistivity of the substrate material. Hence, the heating effect experienced by different substrates will be different. The heating is also dependent on the RF power. Thus, for a consistent heating of the substrate, a controllable heating system that can be embedded into the substrate table will be designed in order to obtain DLC films with reproducible quality.

The use of plasma polymerization in the continuous wave (cw) mode leads to fragmentation of the monomers. The resulting polymer films are strongly cross-linked and chemically different from the respective classical polymer (Han *et al.*, 2000). Since the early 1970s many attempts have been made to achieve plasma polymers with better-defined chemistry by decreasing the energy load. The most suitable method to generate chemically better defined plasma polymer films seems to be the asymmetrically pulsed radio-frequency plasma polymerization, which was established first by Yasuda and Hsu in 1977 (Yasuda and Hsu, 1977), and since then developed further also by other research groups (Han *et al*, 1998; Oehr *et al*, 1999). The rather low power input, that is approximately 10 times lower than that which is characteristic of continuous plasma, combined with a lowered UV damage, which may take place only during the very short plasma impulses, yields polymer coatings that are much more similar to classically produced polymers. Pulsing of the discharge also has the benefits of decreasing the substrate temperature and therefore reduces the stress in the films (Kumar *et al*, 1999), or it can decrease the average power delivered in order to prepare plasma polymers with high monomer structure retention (Han *et al*, 2000). Martin and his co-workers (Martin *et al*, 2004) demonstrated that the morphological characteristics of thin films obtained with pulsed plasma CVD are much better than those obtained with continuous plasma. They explained that these films grow from fewer and smaller nuclei, giving time for the growth processes to cover the surface before new nuclei start a new layer. Therefore, these films have an extremely low roughness (as low as 0.3 nm). However, this kind of growth implies that the overall growth rate is small. This approach would be very interesting to applications such as of photovoltaic coatings, which require very uniform films because it enables better morphological characteristics.

With all these advantages in view, a pulsed PC-RFICP system will be developed for the PECVD (or plasma polymerization) of organic based thin film such as conducting polymer thin films. A schematic diagram of such a system is shown in Figure 5.1. In this system, a sinusoidal 13.56 MHz wave generated by a signal generator is modulated by a pulse wave generated by a pulse generator in order to produce an RF sinusoidal wave signal that periodically turns off. This signal is amplified by a broadband amplifier and then supplied to the center of the spiral induction coil via matching circuit. The other end of the induction coil is electrically grounded. A trigger signal from the pulse generator is sent via a delay generator to the controller of the Langmuir probe. Since the computer can capture data in synchronization with the trigger signal, the plasma parameters can be measured at various times within the pulse period by changing the delay time set by the delay generator.

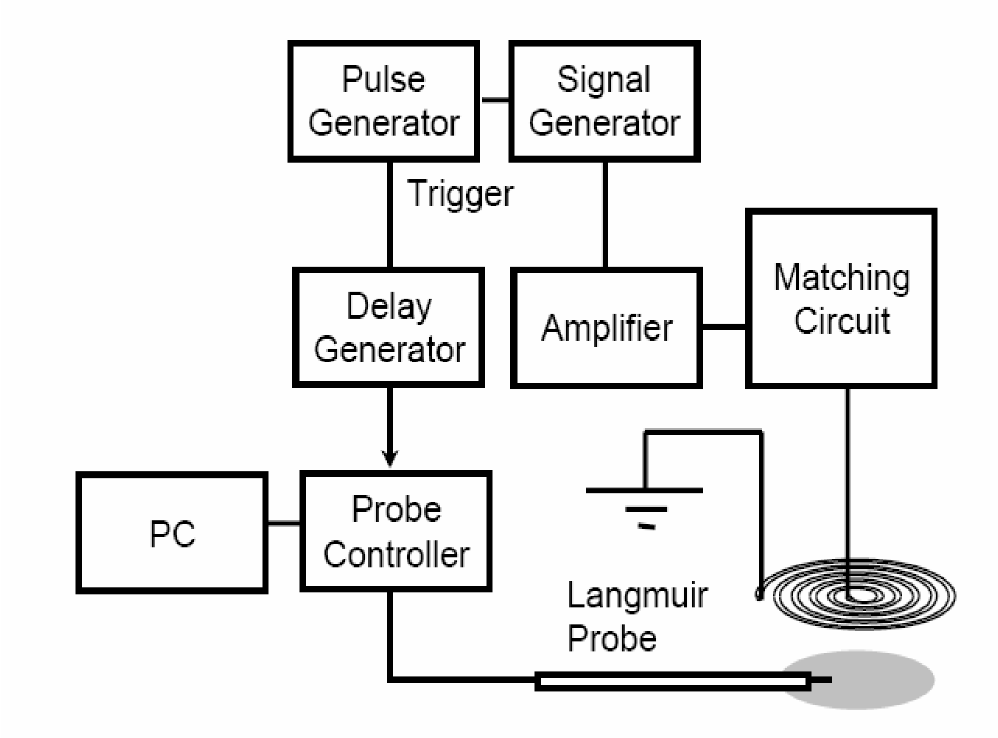

Figure 5.1 Schematic diagram of a pulsed PC-RFICP system.

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